DEPARTMENT OF MECHANICAL ENGINEERING

AHMADU BELLO UNIVERSITY,

ZARIA.

HARDENABILITY RESPONSE OF ALADJA ST 60-2 STRUCTURAL STEEL QUENCHED IN FATTY BASE LOCAL OILS

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TITLE PAGE

HARDENABILITY RESPONSE OF ALADJA ST 60-2 STRUCTURAL STEEL QUENCHED IN FATTY BASE LOCAL OILS

BY

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A thesis submitted to the postgraduate school, Ahmadu Bello University, Zaria, in partial fulfilllment of the requirements for the award of the degree of Master of Science in Production Engineering.

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July, 1991

DECLARATION

I hereby declare that this work is wholly the author's and has not been part of any presentation for any other qualification.

Signature.....

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Date. 16/7/91

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July 1991

CERTIFICATION

This thesis titled "Hardenability response of Aladja ST 60-2 structural steel quenched in fatty base local oil" by Ibrahim Gbadamasi, meets the regulations governing the award of the degree of science (Production Engineering) of Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

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DEDICATION

DEDICATED
TO
THE DEDICATED
TO MY SUCCESS

...THE GBADAMASIS

ACKNOWLEDGEMENT

While this work represents the individual effort of the author, its accomplishment could not have been possible without the assistance and support of several people. It is to them that I wish to record my honest and sincere gratitude.

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ABSTRACT

The suitability of three types of locally produced fatty oils, as quenchants was investigated in this research work. Palm oil, shea butter oil and ground nut oils were used in quenching "ST 60-2" plain carbon steel produced by Delta Steel Company, Aladja. The quench characteristics of the steel as determined by the Jominy End Quench Test compare favourably with the established data for similar grade of steel. Based on the Grossmann test procedure, hardenability of the steel was estimated by measuring the centre hardness of bars with various diameters quenched in these investigated oils.

The quench severity of each oil under investigation was determined using the technique developed by Detroit Diesel Engine division of the General Motors Company (U.S.A.)

Experimental results indicate a strong dependence of the cooling properties of the oils on their chemical compositions. The presence of reactive (Polar) carboxylic group in addition to the fatty acid content of the oils, contribute to strong molecular adhesion of the oils to metal. This adhesion affects the heat extraction properties of the oil.

The iodine content which reflects the degree of saturation is plausibly the most influential chemical constituent which determines quench severity of the oils under investigation.

Palm oil which is rated as the best with a quench severity (H) value of 0.12 - 0.15 is capable of generating a hardness value equal to that of 50% martensite at the centre of a bar of 10.2mm maximum diameter as revealed by the Grossmann test. Shea butter and groundnut oils gave similar result with quench severity values of 0.08 - 0.10 and a hardenable bar diameter of 9mm.

Microstructural examination of the quenched bars were further used to corroborate hardness measurements.

Microstructural examination revealed, principally, ferrite - pearlite structure in all the quenched steel rounds. The observed increase in hardenability in quenched steel rounds in various media can be attributed to the formation of finer and, therefore, stronger ferrite pearlite structure. Definitely, the steel will respond favourably to alloying elements for martensite formation.

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ABBREVIATIONS

q_x	=	Heat transfer rate in the x-direction (W)
A	=	Area normal to direction of heat flow (m^2)
dT/dx	=	Thermal gradient in the X-direction (K/m)
K	=	Thermal conductivity (W/m.k)
β	=	Thermal diffusivity
∇^2	=	Laplacian operator
t	=	Time
x,y,z	=	Cartesian coordinates
r,R	=	Cylinder radius
λ_m	=	Roots of Bessel functions
h	=	Coefficient of corrective heat transfer (W/M^2k)
T_o	=	Surface temperature of metal at time = O
T_s	=	Temperature of surrounding
T_{axis}	=	Temperature at the axis of the metal
T	=	Temperature of metal at time t
$\emptyset$	=	Function
H	=	Quench severity
F_o	=	Fourier modulus
Bi	=	Biot Modulus
ΔT (To-Ts)	=	Temperature difference between metal surface and fluid (surface temp. excess)
q	=	Rate of convective heat transfer
L	=	Length (m)
Wvx	=	Vapour velocity
W_x	=	Fluid velocity
δ_{vx}	=	Vapour thickness
σ	=	Surface tension of vapour / liquid interface
R_b	=	Radius of bubble
ρ_L	=	Density of Liquid at saturation temperature
ρ_p	=	Density of vapour at saturation temperature
dT/dP	=	Temperature variation with vapour pressure.
P_1	=	Vapour pressure over flat surface
P_2	=	Vapour pressure of bubble
N_{NU}	=	Nusselt number
C	=	Constant
N_{RE}	=	Reynolds number
N_{Pr}	=	Prandtl number
DPN	=	Hardness vickers
P-oil	=	Palm Oil
G/Nut oil	=	Ground nut oil
S/Butter	=	Shea Butter oil
C/Water	=	Cold water
H/Water	=	Hot water.

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CHAPTER I

1.0 INTRODUCTION

1.1 GENERAL

Nature provides about 50 metallic elements; by alloying these elements, the number of theoretically possible alloys reaches approximately 400,000 (USS, 1966). However, technological and economic considerations limit choice to a few basic metallic elements such as Fe, Al, Ni,Ti, etc, and a small number of alloying elements.

Most of the progress made in the technical field can be closely linked to the advancements made in the development of materials. Alloys, such as steel or iron castings have been in use for centuries and remain the most important materials for many industrial applications (Beck, 1981). Despite these widespread applications of alloy steels and cast iron, technological advancements have put more stringent demands on improved quality and enhanced properties. Improvements in properties are, however, chiefly acquired by controlling composition and by further processing in the form of heat treatment and mechanical working.

Steel, as an engineering material, is versatile; this versatility arises from the fact that its properties are very amenable to heat treatment. Heat treatment principle governs the procedure required to obtain particular microstructure in a given metal to suit desired working conditions. The procedure involves heat transfer processes in the solid and between the solid and its surrounding. A review of the

various stages involved in these treatments shows that quenching is the most critical one.

The desire to obtain optimum properties in these steels economically requires that the nature of the factors involved in the processing of the material are thoroughly understood so as to facilitate the design of alloys which conform to desired specifications. Based on the type of applications, majority of steel components require specific heat treatment procedure to produce structures having both the chemical and mechanical properties needed under such applications. In developing Countries, where these alloys are not availaible, it is then necessary to upgrade the use of already existing alloys.

Metastable structures such as that in quenching are as a result of fast cooling of a piece that has been previously raised to a high temperature (850 - 1200 ^{o}C). While fast cooling can be realized by quenching in a vapourizable liquid such as water, mineral oil or a solution of organic polymers (Moreaux and Beck, 1981), it can also provoke high thermic gradients inside the parts resulting in dilatation differences between different zones of the metal. From a general point of view, treatment by quenching are expected to achieve the following:-

(i) Provide a fast cooling of the part to give good and desirable microstructure and

(ii) The cooling must be such as to minimize thermal gradients and the consequent damaging of parts.

In this research, the concept that considers steel to have a base hardenability derived from the carbon content and austenite grain size (Lesile, 1982) is being exploited. Plain carbon steel produced in Nigeria was thus used to assess heat extracting properties of some Nigerian fatty oils in quenching operations. Specifically, it seeks to investigate alternatives to water and mineral oil quenchants. In essence, preliminary investigations on the use of some locally produced fatty base oils as quenchants for plain carbon steel produced in Nigeria are being conducted in the present research effort.

1.2 Heat Treatment

The science of heat treatment deals with the factors and mechanism involved in the control of the constitution of an alloy by heating and cooling in the solid state, and the relationship between the constitution and properties of the alloy. In steel, it is generally taken to embrace the martensitic transformation which occurs on quenching from the austenitic state. Depending upon properties desired, a tempering operation may also be effected.

1.2.1 Heat Treatment Procedure

The first step in heat treatment process is the heating of the material to a temperature at which austenite is formed. This temperature is such that all carbides are in solution so as to homogenize the piece but not to promote excessive grain growth. The mass and shape of the material

determine the heating rate and also the rate the material can absorb heat (USS, 1971). The usual safe practice is to slowly and uniformly heat to the austenitizing temperature followed by a holding period at that temperature. A heating schedule based on percentage carbon content of the steel, as given in table (1.1), is usually recommended (Lesile, 1982).

On completion of austenitization, the piece is then subjected to a cooling schedule as to induce transformation of the material to give desirable microstructural constituents.

Table 1.1 Schedule for Normalizing and Austenitizing Temperatures for Plain Carbon Steel

CARBON CONTENT	NORMALIZING	AUSTENITIZING
Up to 0.25 %C	925 + 5 °C	925 + 5 °C
0.26 to 0.36 %C	900 + 5 °C	870 + 5 °C
0.37 and over	870 + 5 °C	845 + 5 °C

Quenching is the heat treatment commonly used to induce hardening effect in the material. The resulting microstructural constituents, theoretical lower banite and martensite, depend on the transformation rates which must be slow enough to prevent prior transformation at a high temperature during quenching. The martensite is a super saturated unstable alpha-iron solid solution (Bain and Paxton, 1966).

The usual practice is to subject the material to a prior normalizing treatment before it is quenched. Normalizing, primarily serves two purposes: to refine the grain and to

obtain a carbide size and distribution which will be more favourable for carbide solution on subsequent heating than the as-rolled structure. It involves heating the steel above its critical temperature and air cooling.

In general, the suitability of a steel for a given heat treatment practice or the suitability of a heat treatment practice for a given steel is determined, largely, by its hardenability the measurement of which serves to establish the limiting conditions in terms of cooling rates or quenching practices necessary to obtain the desired microstructure.

1.2.2 Austenite Transformation In Quenching Operation

Quenching involves controlled super-cooling of austenite which transforms to a wide range of possible structures and properties. Under equilibrium conditions (slow cooling), austenite transforms to ferrite and pearlite. However, when more rapid cooling is employed, as is the case in quenching operations, this transformation is depressed and the resulting microconstituents differ.

The two constituents of steel, the amount and distribution of which primarily control the properties, are Iron (ferrite) and iron carbide (cementite). While cementite is not found as the matrix (principal phase), it exerts a profound influence on the performance of the steel (Lesile, 1982). Usually, cementite is found embedded either as a

network with an infill of pearlite or as spheroid in ferritic or martensitic matrix. Ferrite is used to include any solid solution in alpha-iron such as austenite (Pascoe, 1978).

Formation Of Microstructures

Quenching, unlike slow cooling, is not diffusion controlled, and as such, the microstructural constituents that result are not determined by the nucleation and growth mechanism. Bain and Paxton (1966) reported that nucleation and growth times determine the formation of ferrite and pearlite. Though nucleation rates increases rapidly with decreasing temperature of transformation, growth rate does not vary by more than a factor of five between 700 ^{o}C and 600 ^{o}C (the eutectoid transformation temperature range, that is, Austenite $\rightleftharpoons$ ferrite and cementite). Since growth is controlled by diffusion of carbon that is rejected into the austenite, the size of the ferrite grains increases only with the square root of time. They state that nucleation of pearlite which only forms just below the temperature, 700 ^{o}C - 600 ^{o}C, is slow compared to that of ferrite while the growth rates are similar.

However, at lower temperatures, the nucleation time for pearlite approaches that for ferrite and the growth rate of pearlite at 600 ^{o}C is about 50 times as high as at 700 ^{o}C. Thus, although ferrite may begin to grow slightly before pearlite when the pearlite interruption takes place, nearly all the remaining austenite is consumed by pearlite encroachment.

In plain carbon steels, high possibility of ferrite formation exists before austenite transforms to pearlite (Wilson, 1973). However, the maximum possible amount of such constituents (ferrite) is controlled by the rate of cooling, since the reaction is essentially one of diffusion, and such diffusivity is a function of temperature. So the greater the degree of undercooling, the smaller the size of ferrite formed. If, however, the pearlite formed is such that it has fine lamellar or accicular structure, it is termed banite. Pearlite will not necessarily grow at exactly eutectiod composition according to Bain and Paxton (1966). However, they reported that properties do not depend on composition as much as on the carbide spacing, which is, in turn, a function of the temperature of formation of the pearlite.

When the steel are quenched at a rate exceeding the critical cooling rate, the transformation which is rapid starts to occur at a temperature near 230 ^{o}C (Bain, 1966).This results in the formation of martensite.

This structure does not involve nucleation and growth and requires no atomic diffusion. Welty et al. (1976) suggest that it is based on a shearing process which involves large parts of the parent crystal. The structure is characterized by accicular or needle - like pattern and it is hard and brittle. Thus the structures in quenched carbon steel can be classified as:

(a) Lamellar (Pearlite, Banite) and

(b) Martensitic

The first family of structures form above 550 ^{o}C on a not-too-rapid cooling. The second family results from rapid cooling and a transformation usually not far above room temperature (Bain, 1966).

1.3 Concept Of Hardenability

The most important characteristic of heat treatable steel is that which determines the amount of martensite produced under a given rate of quenching. This characteristic is termed hardenability.

Hardenability is the ability of a steel to form 100% martensite on cooling from the austenite range as reflected by its critical cooling rate. (Welty et al 1976). McGannon (1971) defined hardenability, in practical terms, as that size of a piece which can be hardened under given cooling conditions.

The critical cooling rate is a measure of how slowly the steel can be cooled without transforming, even partially to proeutectoid ferrite or carbide, pearlite or Banite (Bain, 1976). This rate depends upon the carbon content, the relationship of which can be represented as schematically shown in figure 1.1 (Pascoe, 1978).

It follows from the definition of Bain and Paxton (1966) that, if the cooling rate is less than the critical cooling rate, the structure may contain some martensite together with either ferrite or pearlite, or Banite, or a mixture of these.

This attribute of steel, hardenability has two-fold significance according to McGannon (1971): it is important

in relation to the attainment of a higher martensitic hardness or strength level by heat treatment and also in relation to attainment of a high degree of toughness through heat treatment.

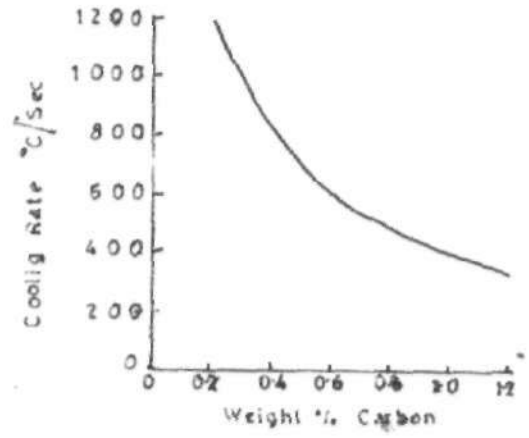


Fig 1.1: Variation of Critical Cooling Rate with Carbon Content of Carbon Steels

The improvement of hardness is, essentially, an enhanced capacity to develop the characteristic martensitic hardness for the steel with a slow cooling rate; this is independent of the intrinsic hardness of the martensite. The intrinsic hardness is dependent almost entirely upon the carbon content of the steel while the hardenability is, in general, far more dependent upon the alloy content and grain size of the austenite.

The second factor,(attainment of desirable microstructure for improved toughness) depends largely on the rate of cooling (severity of quench) that will induce substantially martensitic microstructure; for example, a steel bar of a particular composition may harden completely

in an oil quench, while a similar bar of different composition will require a more drastic water quench and even then, only the surface layer may escape transformation to the softer high temperature structure. The case of drastic quench manifests in low-carbon unalloyed steels where shallow martensitic depth is obtained but with considerable ductility and toughness due to transformation of the remaining austenite to other products usually banite and/or pearlite. This same effect results when a fairly large mass of steel is quenched; the core cools less quickly than the outside, resulting in only part (from the surface) of the piece been of martensitic structure. This feature is referred to as the mass effect.

In general though, only a small proportion of carbon or alloy steel reaches service in the martensitic state; however, their mechanical properties are usually of no prime consideration if they do.

1.3.1 Assessment Of Hardenability

(a) Critical Cooling Rate:

Since hardenability is applied to the particular property of steel which determines their ability to transform to a substantially martensitic condition on cooling at some minimum rate, the minimum cooling rate that is the critical cooling rate, is then the most direct measure of hardenability which can be used to assess various quenchants.

(b) Depth of Hardening:

Hardenability is manifest also by the failure of large bars to harden throughout and, instead, in a specific quenching procedure, to harden only in the outer zone. Thus, the penetration of a certain degree of hardening in uniform-sized similarly quenched specimens is another measure of hardenability. This relies on the "heat extraction qradient" across a cooling mass where it has been established (Wilson, 1975) that the interior cooling rate is slower.

(c) Cylindrical Bar Test:

Here, hardenability manifestation is in the size of a cylindrical piece that will just exactly harden through with a given quenching procedure. The limiting diameter of a suitable round then becomes a measure of hardenability.

The method of critical cooling rate has the disadvantage that in practice, cooling rates are ordinarily not constant, but vary during the cooling cycle. This is particularly true of liquid quenching in which case, the cooling rate is always slower as the temperature of the cooling medium is approached (McGannon, 1971). Furthermore, means of correlating cooling rate and depth of hardening that defines hardenability, is required.

The other two tests (depth of hardening and cylindrical bar test) require hardness value measurements and/or microscopic examination to determine points and limit of the formation of martensite. It is also required that, under these tests, bars of given length are employed such that a section, approximately midway between the ends are not

subject to end cooling effects. Grossmann et al (1930) employed a criterion in which martensite forms more than 50% of the microstructure at the centre after a certain quench. The reason adduced to this choice by Bain and Paxton (1966) was that the transformation zone, particularly for shallow hardening steels, composed of a mixture of martensite and fine pearlite, is narrow.

They agreed that, since rapid change in hardness occurs in the range between 40% and 60% martensite, the choice of 50% was an excellent criterion. Thus, this method not only gives depth of hardening, it expresses it with respect to the characteristic of quenchant used, which in turn, can be compared to standard quenching medium of known quench severity.

1.3.2 Factors Affecting Hardenability

The final microstructure of a quenched steel, and therefore the properties are largely dependent upon the transformation behaviour of the austenite and on the cooling conditions. Thus, hardenability or the extent of formation of martensite of a piece can be predicted if these factors are known or can be governed by controlling either or both of these factors.

In general, any change in the steel that delays and retards the transformation of the austenite in the range of its maximum transformation temperature rate contributes to slower critical quenching speed and in turn to its hardenability. Thus, hardenability which relates to the

minimum quenching rate that will produce martensite and permit the steel to escape transformation to softer constituents controls the size of section and also the depth of hardening in larger sections of homogeneous chemical composition. In effect, a material with higher hardenability will form martensite in larger sections on quenching.

Hardenability is altered by factors other than mass of the steel and the rate of cooling. Fundamentally, three major factors, intrinsic to the steel, influence hardenability; these are: austenite grain size, homogeneity and composition (McGannon, 1971).

(a) Austenite Grain Size:

Austenite grain size, at time of quenching, exercises control over nucleation capacity which takes place at grain boundaries. Thus, the greater the grain boundary area per unit volume, the greater will be the density of nucleation sites. Consequently, fine grains with greater grain boundary surface would likewise increase the probability of nucleation and reduce hardenability. Grange (1973) observed a linear relationship between the diameter of shallow hardening steels hardenable to 90% martensite and the inverse square root of grain diameter.

(b) Homogeneity:

Inhomogenity of austenite speeds up the start of transformation thereby resulting in fine pearlite. The mechanism is by initiation of transformation in the portions of the austenite which are lean in alloy and/or by the process of undissolved carbides acting as nuclei for trans-

formation, thus lowering hardenability in these areas. Bain and Paxton (1966) gave vanadium as a classic example of special carbide forming elements.

(c) Composition:

Steels of varying carbon and alloy composition harden to different degrees when quenched. Houghton (1960) observed that increase in carbon content and alloy content, except in the case of cobalt, generally tends to decrease transformation rate and/or increase the time for its completion, hence, improve hardenability.

Based on the procedure developed by Grossmann et al, Lesile (1982) reported that the overall effect of different alloying elements on hardenability can be calculated by multiplying the presumed base hardenability derived from carbon content and grain size with certain factors for each alloying element present. The factor varies with the effectiveness of the element in increasing hardenability and the concentration of the element. It was ascertained by McGannon (1971) that the effect of alloy additions is cumulative. Houghton (1960) classified the elements into two broad groups. The first group is made up of those elements that retard both pearlitic and intermediate compound reaction (e.g boron, copper, manganese, nickel and silicon). The other group consists of those that mainly retard the pearlitic reaction (e.g chromium and molybdenum). The "adverse" effect of cobalt was explained by Bain (1966) as being due to the increase in free energy change serving as

driving force during transformation of cobalt containing austenite.

Increase in the carbon content up to the eutectoid composition retards the pearlitic reaction; beyond this, the incubation period is decreased causing a quickening of the rate of reaction.

Siebet et al (1977) reproduced the relationship between the percentage carbon composition and hardenable diameter for carbon steels schematically shown in figure 1.2

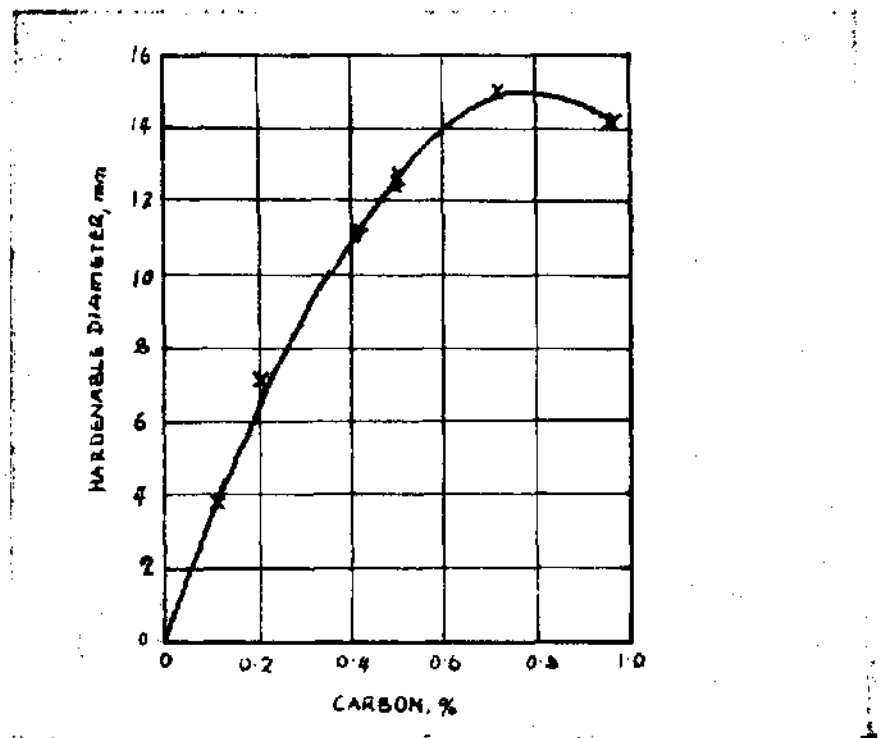


Fig. 1.2: Hardenability of FC-C Alloys (90% Martensite) Water Quench

In summation, the following factors decrease hardenability: (Bain and Paxton, 1966)

(i) Fine grains of austenite

(ii) Undissolved inclusions:

- Carbides or Nitrides

- Non metallic inclusions

(iii) Inhomogeneity of austenite

While the following factors increase hardenability:

(i) Dissolved elements in austenite (except cobalt)

(ii) Coarse grains of austenite

(iii) Homogeneity of austenite

(d) Factors Extraneous to the Steel:

In a number of practical treatments, properties intrinsic to the steel are usually separated from the conditions of the quench so that the latter may be represented by the factor H. (quench severity). This was the basis of the work of Grossmann et al (1930). As a result of the surface reactions which occur when a piece comes into contact with the quench, several factors influence the transmitted heat flux in the boundary layer of solid/liquid and consequently the rate of cooling and thus the hardenability of the material. Amongst several others, agitation of the media, the heating technique of the part, and temperature of the quench are factors that must be stabilized to obtain good hardening.

1.4 Material Selection For Hardenability Test

In small sections, carbon steel may be quenched to develop martensite. To secure these superior properties without going to considerable trouble, and to secure them, not only in small section or near the surface of moderate section, but throughout large sections, requires the addition of alloying elements. Customarily, hardening of steel is considered from the point of view of the realization of full martensitic hardness. In practice, however, prevention of the transformation of austenite to lamellar structures is the primary function of quenching and a most important reason for the use of deep-hardening alloys.

Economic consideration in respect of the cost of these alloys and their shortage has lead to the reappraisal of carbon steels. This has also initiated need for refinement in heat treatment methods and careful selection of cooling rate as a means of adjusting or attaining higher properties from carbon steel.

1.4.1 Steel Selection

Properties Of Carbon Steel

Plain carbon steels may be defined as steel containing up to 1.66% Mn, 0.60% Si and 0.60% Cu in addition to much smaller amounts of other elements and the percentage carbon not exceeding 1.0%. About 50 grades (Metals Hand Book 8th Ed, 1961) are available in the non-resulphurized series.

Properties of carbon steels depend chiefly on their carbon content and microstructure. Their properties may be

modified by the effect of residual amounts of Mn, Si, P and minor amounts of alloying elements or the presence of gasses, especially, oxygen, nitrogen and hydrogen. The final properties of these steels are therefore influenced by the steel making practice used in their production.

Method of production then serves as one of various ways of classification of carbon steels. They can be open hearth, basic oxygen or basic electric furnace, or by extension to include deoxidation practice employed such as rimmed, capped, semi-killed or fully killed steels. Another method of classification given in the American Society for Metals Hand Book 8th Ed (1961) is based on carbon content as follows:

(a) 0.10 to 0.25% Carbon:

Here, treatments as to induce hardening is not cost justifiable and thus used only to improve machinability and to obtain a black surface finish commercially accepted. Thus they are only giving conditioning treatments for further processing.

(b) 0.25 to 0.55% Carbon:

Steel of this group are the most versatile because of high carbon content. They are used in hardened and tempered condition, usually for couplings, tie rods, crank-shaft etc. Within this group, there is a continuous change from water hardening to oil hardening types, thus quenching media include water, caustic soda, brine and oil.

(c) 0.55 to 1% Carbon:

Application of these are more restricted because of higher carbon content which results in difficulty to

fabricate. They are also more brittle in heat treated condition and are used in nearly fully hardened state (Rockwell C hardness value 55) where abrasion resistance is of prime importance.

Carbon Steel For Hardenability Test

Carbon steels have a relatively low hardenability because their austenite transformation behaviour is determined almost entirely by the carbon and manganese content as opposed to alloy steels wherein alloying elements aid hardenability.

The maximum carbon content of the non-resulphurised carbon steel series for direct hardening is often placed at about 0.25% C and have 0.6 - 0.9% Mn content (Metals Hand Book 8th Ed, 1961). Increasing the carbon and manganese content improves hardenability up to about 0.4%C when the steel become susceptible to quench cracking (Siebert et al 1977). Despite the fact that these steels have shallow hardenability, the danger of cracking still exists when the part is subjected to excessive drastic quenching.

Carbon steels produced in Nigeria which conform to the above composition of direct hardening steels are being used in this investigative work. This eliminates the need to take into consideration the effect of alloying elements, thus enhancing direct assessment of carbon steel with respect to the oils under investigation.

1.4.2 Selection Of Quenching Media

The first requirement of a quenching medium is a sufficient cooling rate to achieve martensization. The cooling potential of the quenching medium is a measure of quenching severity and can be varied over a wide range by:

(a) Selection of a particular quench

(b) Control of agitation and

(c) Additives to improve cooling power of the quenchant.

The most commonly used quenchants are water, oil or brine. The relative severity of quench of these media (table 1.2) rates brine as having the highest severity while oil is considerably less drastic; water quenching is less costly than oil quenching, however, the degree of distortion and cracking that occurs in water quenching can be quite high.

Steels to be oil quenched require alloy content to provide the hardenability required.

In light of relative advantages and disadvantages of oil, brine and water, this research work sets out to investigate the adaptability and use of some fatty oils obtained in Nigeria as quenching medium with the following advantages being sort.

(a) Permit use of less-expensive (lower alloy) steels of lower hardenability.

(b) Optimize properties of the steel selected.

(c) Permit use of less-expensive quenching medium.

(d) Improve productivity and achieve cost reduction as a result of shorter cycle times and higher production rate.

TABLE 1.2 Approximate Values of H (Severity of Quench)

AGITATION	OIL	WATER	BRINE
NONE	0.25 - 0.30	0.9 - 1.0	2
MILD	0.30 - 0.35	1.0 - 1.1	2.0 - 2.2
MODERATE	0.35 - 0.40	1.2 - 1.3	-
GOOD	0.40 - 0.50	1.4 - 1.5	-
STRONG	0.50 - 0.80	1.6 - 2.0	-
VIOLENT	0.80 - 1.1	4 - 0	5.0

CHAPTER II

LITERATURE REVIEW

2.1 Mechanism of Quenching

The quenching of steels involves unsteady state heat flow because it involves variation of temperature with respect to time, position, and solid state transformation (Gary, 1975). The interdependence of these factors complicates prediction of phase transformation in metal heat treatment processes such as quenching.

Heat may be transmitted in different ways; by conduction, convection or radiation. Conduction and convection modes of heat transfer dominate when a solid is quenched in a fluid which is at a lower temperature.

Conduction and radiation are the two basic and fundamental different mechanism of heat transfer. Radiation involves the propagation of energy through space at the speed of light and, like the transfer of lightened radio waves, is an electromagnetic phenomenon (Grober and Erk, 1961). In short, radiation is the energy transported in the absence of matter. Conduction, on the other hand, necessarily requires the existence of matter and depends on the molecular activity within the matter.

Thermal convection is not a basic heat flow mechanism like radiation and conduction. It mainly involves conduction of heat to the exterior surface of the solid being cooled together with the physical movement of the fluid surrounding the cooling body. Convection may be correctly described as the activity of surface conduction because the heat flow from

a cooling surface to the surrounding fluid is governed by the conduction process through a thin layer of fluid adjacent to the surface.

Convection heat transfer is subdivided into forced convection where the fluid is forced over the surface by a blower or pump, and natural convection. These processes may also involve phase changes within the fluid around the cooling solid; these leads to the important fields of boiling and condensation convective heat transfer processes.

2.2 Heat Transfer During Quenching

Any physical phenomenon is generally accompanied by a change in time and position of its physical properties. The process of heat transfer takes place as a result of difference in temperature at different positions of the body. In quenching, this is accompanied by the dissipation of heat to the surrounding.

Quenching operation involves propagation of heat through the solid by conduction followed by convection in the fluid.

Energy transfer by conduction is accomplished in two ways (Welty, et al 1976); molecular interaction within the medium or free electron movement. Conduction by molecular interaction is that in which the greater motion of a molecule at a higher temperature imparts energy to adjacent molecules at lower energy levels. This type of transfer is present, to some degree, in all systems in which a temperature gradient exists and in which molecules of a

solid, liquid or gas are present. The free-election mechanism is significant primarily in pure-metallic solids.

However, thermal conduction in the solid phase is attributed both to molecular interaction and to free electrons. Since mathematical theory disregards the molecular structure of matter and considers matter to be continuous (Grober and Erk, 1961), it then follows that, the ability of solids to conduct varies directly with the concentration of free electrons. The first basic equation used to describe the process of heat conduction was given in 1822 by Fourier in the form

$$q_x/A = -k \frac{dT}{dx} \quad \text{---------- (2.1)A}$$

q_x/A is defined as the heat flux in x-direction

The general form of this basic equation is expressed as follows:

$$q/A = -k \nabla T \quad \text{----------(2.1)B}$$

It expresses the heat flux as being proportional to the temperature gradient for an isotopic medium. This is referred to as the "Fourier Rate Equation" (Isancheko, et al 1974).The constant K is the thermal conductivity and represents the ability of a material to transport heat.

The thermal conductivity is a material property. It drops in the presence of all kinds of admixtures because of increase in structural heterogeneity. However, in contrast to pure metals, the thermal conductivity of alloys increases with rising temperature making it primarily a function of temperature (Isachencko et al, 1974).

By considering changes in internal energy, a more general form of the Fourier equation was obtained as follows:

$$dT/dt = -k/c\rho \left[d^2T/dx^2 + d^2T/dy^2 + d^2T/dz^2 \right] \quad \ldots\ldots\ldots\ldots(2.2)A$$

that is

$$dT/dt = -B \nabla^1 T \quad \ldots\ldots\ldots\ldots(2.2)B$$

This is called the Fourier field equation. The factor of proportionality, β (thermal diffusivity) is a measure of the thermal inertia of the solid.

Since dT/dt is proportional to β, it follows that the rate of change in temperature at any point of the solid is high for large thermal diffusivity. Unlike metals, liquids and gases posses a higher thermal inertia and hence a smaller thermal diffusivity.

The fourier field equation [(2.2)B] describes the phenomenon of conduction in its general form. To give a comprehensive description of any physical situation, the prevailing characteristic condition of the test will dictate the system boundaries. The initial and boundary conditions, together with the differential equation, gives the solution to energy transfer by conduction.

Initial Conditions:

This refers specifically to the law of temperature distribution within the body at the initial moment of time. They are given for the study of transient processes and are described analytically as:

$$T = f(x, y, z) \quad \text{for } t = 0 \quad \ldots\ldots\ldots(2.3)$$

Where temperature distribution is uniform, the initial condition is simply:

$T = T_O$ = constant for $t = 0$

or $T/_{t=o} = To$ = constant

Boundary Conditions:

This refers to the value of T and x, existing at specific positions on the boundaries of a system.

2.2.1 Test Condition I

Conduction in Immersion Quenching

Conduction of test is such that an infinitely long cylinder steel piece at a uniform temperature T_O, is placed in a new surrounding with its surface suddenly and simultaneously exposed to the fluid at temperature T_s.

The Fourier field equation, [(2.2)B] can be expressed in cylindrical co-ordinates thus:

$$dT/dt = -\beta \left[d^2T/dr^2 + 1/r\ dT/dr + 1/r\ d^2T/d\phi^2 + d^2T/dz^2 \right] \quad \ldots\ldots\ldots\ldots(2.3)A$$

Since the cylinder is assumed infinite in length and symmetrical with respect to the axis, the temperature distribution will be independent of Ø and Z directions, thus equation [(2.3)A] reduces to

$$dT/dt = -\beta \left[d^2T/dr^2 + 1/r\ dT/dr \right] \quad \ldots\ldots\ldots(2.3)B$$

Applicable conditions are:

Initial Condition:

(1) $T/_{t=o} = T_O$ = constant

(2) $(dT/dr)_{r=o} = 0$ at r=0 for t > 0

Boundary Condition:

$$(dT/dr)_{r=R} = -H\,(T - T_S) \qquad \text{for } t > 0$$

Solution to equation [(2.3)B] involves separation of variables, integrating the equation and then determining the integration constants from the initial and boundary conditions. The solution was given by Welty et al (1976) as:

$$\frac{T - T_S}{T_O - T_S} = 2\sum_{m=1}^{\infty} \frac{e^{-\lambda_m^2 \beta t}}{\lambda_m} \frac{J_O(\lambda_m r/R)\, J_1(\lambda_m)}{J_O^2(\lambda_m) + J_1^2(\lambda_m)} \qquad \text{.....(2.4)}$$

By substituting r=R and r=o, equation for the temperature at the surface and axis respectively can be obtained thus:

For the surface

$$\frac{T - T_S}{T_O - T_S} = 2\sum_{m=1}^{\infty} \frac{e^{-\lambda_m^2 \beta t}}{\lambda_m} \frac{J_O(\lambda_m)\, J_1(\lambda_m)}{J_O^2(\lambda_m) + J_1^2(\lambda_m)} \qquad \text{.....(2.5)}$$

For the axis

$$\frac{T - T_S}{T_O - T_S} = 2\sum_{m=1}^{\infty} \frac{e^{-\lambda_m^2 \beta t}}{\lambda_m} \frac{J_1(\lambda_m)}{J_O^2(\lambda_m) + J_1^2(\lambda_m)} \qquad \text{.....(2.6)}$$

Isancheko et al (1974) gave a graphical illustration of the time variation of temperature within the cylinder as shown in figure 2.1 below:

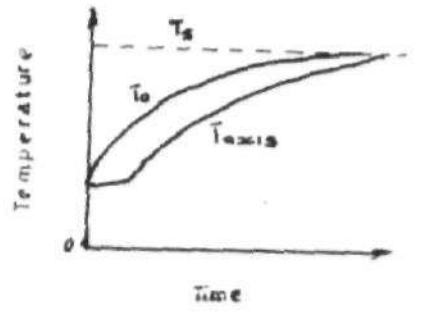


Fig. 2.1 Time- Temperature Variation Within a Body
T = F (t)

The solution as obtained in equations (2.4) to (2.6) involve Bessel functions. The factor λ_m are roots obtained from the relation.

$$(\lambda_m R)\ J1\ (\lambda_m R) - HR\ J_O\ (\lambda_m R)$$

which are usually tabulated as found in Carslaw and Jaeger (1959).

Welty et al (1976) gave a simpler form of the equation by representing the solution in "Bachman's dimensionless variable" thus:

$$\frac{T - T_s}{T_o - T_s} = \emptyset\ (\ hR/K\ ,\ Bt/R^2\ ,\ r/R\ ) \quad .....(2.7)$$

where
Bt/R^2 is termed the Fourier modulus (Fo)
and hR/K is the Biot modulus (Bi)

The Biot modulus is the ratio of the conductive (internal) resistance to the convective (external) resistance to heat transfer.

During cooling, the rate of energy transfer is largely dependent upon both internal and surface resistance, the limiting cases being represented either by negligible internal or surface resistance. The over-riding consideration in quenching is then the value of the Biot modulus (Carslaw and Jeager, 1959). Large values of Biot number indicates that conductive resistance is the rate determining process; that is, there is more capacity for heat to leave the surface by convection than to reach it by conduction. The reverse holds true with a small value of the Biot number.

2.2.2 Test Condition 2

Conduction in End Quench Test:

The cylindrical test piece, initially at a uniform temperature T_o, is exposed at one end to a vertical jet of water at constant lower temperature T_s. The procedure is such as to approximate a unidirectional heat conduction in the material. Since the rate of conduction along the bar is far greater than the radiative or mild convective radial heat transfer, the condition of unidirectional heat flow is satisfied (Grober and Erk, 1961). The procedure is in conformity with the Jominy End Quench test.

The Fourier field equation as given by Carslaw and Jeager (1959) is

$$dT/dt = \beta\, d^2T/dx^2 \quad \text{---------- -} \quad (2.8)$$

Initial Condition:

$$T\big/_{t=o} = T_S = \text{constant}$$

Boundary condition

$$T\big/_{x=o} = T_S$$

The differential equation is solved with the aid of error functions and the solution given by Grober and Erk (1961) is:

$$T = T_S = \frac{2(T_O - T_S)}{\Pi^{1/2}} \int_0^{\frac{x}{2(\beta t)^{1/2}}} e^{-z^2}\, dz \qquad \text{.....(2.9)A}$$

where

$$\frac{2}{\Pi^{1/2}} \int_0^{\frac{x}{2(\beta t)^{1/2}}} e^{-z^2}\, dz = \text{erf}\left[(x/2\ \ (Bt)^{1/2}\right]$$

and is referred to as the probability integral or error function and has the following properties:

$$\text{erf}(0) = 0, \qquad \text{erf}(\infty) = 1, \qquad \text{erf}(-x) = -\text{erf}(x)$$

So

$$\frac{T - T_S}{T_O - T_S} = \text{erf}\left[(x/2\ \ (Bt)^{1/2}\right] \qquad \text{.....(2.9)B}$$

In Bachmans dimensionless parameter

$$\frac{T - T_S}{T_O - T_S} = \phi\ (Bt/x^2) \qquad \text{.....(2.9)C}$$

The heat transfer is thus a function of dimensionless time (Fo), which implies that the time for two points to reach a given temperature is a function of the square of their distances from the boundary.

2.2.3 Convective Heat Transfer

Heat transfer due to convection involves the energy exchange between the quenched piece and the quenching medium. It is due to associated change and buoyant effect as a result of heating between the layers of molecules in contact with the quenched piece and those further away. This effect produces "natural" circulation (convection) currents. The speed of the currents increases with a larger temperature difference between the piece and the liquid (Greogory and Simon, 1963). Agitation, however, improves the convection process.

The rate equation for convection heat transfer was first expressed by Newton in 1701 and it is referred to as the "Newton Rate equation". The equation is of the form

$$\frac{q}{A} = h \Delta T \qquad \ldots\ldots\ldots\ldots\ldots \quad (2.10)$$

The coefficient of heat transfer h varies with the mechanism of fluid flow, the properties of the fluid and the geometry of a specific system (Welty et al, 1976).

By analogy to the "hydrodynamic boundary layer", Isancheko and co workers (1974) defined a thermal boundary layer where heat is first transferred from the quenched metal to the liquid through a stagnant fluid layer. Mechanism of heat transfer through this layer is that of conduction. The "film" of fluid often represents the controlling resistance to convective heat transfer and the coefficient h is referred

to as the film coefficient (Welty et al, 1976). They gave the rate equation as follows:

$$- q_y = - KA \, d/dy \, (T - T_S)\Big|_{y=o} \qquad \text{.....(2.11)}$$

for thermal boundary layer conduction

$$q = hA \, (T_S - T_\infty) \qquad \text{.....(2.12)}$$

for convection

Equating and introducing a length parameter gives:

$$\frac{hL}{K} = \frac{d(T_S - T)/\, dy\Big|_{y=o}}{(T_S - T)/L} \qquad \text{.....(2.13)}$$

where L is the length parameter.

The factor hL/k is the Nusselt number and is similar to Biot number. It represents the ratio of conductive thermal resistance of the fluid.

However, the nature of convective heat transfer encountered in quenching is characterized by boiling. Welty et al, (1976) reported that the film coefficient, h, associated with the phenomenon of boiling, is of the order of 2,500 - 25,000 W/(m.k) for water; about 3 to 10 times of that found in convection without boiling for the same medium.

2.2.3.1 Boiling Phenomenon In Quenching

Boiling heat transfer is associated with a change in phase, characteristically that of vapourization. It achieves high heat transfer rates while accompanying temperature differences may be quite small. The phenomenon, according to Isancheko et al (1974), is affected by surface tension, latent heat of vapourization, surface characteristics of the metal and other properties.

Grober and Erk (1961) classified boiling into two basic types: pool boiling and flow boiling. Pool boiling is that which occurs on a heated surface submerged in a liquid pool which is not agitated and flow boiling occurs in a flowing fluid. Pool boiling obtains in quenching operations. It originates as a result of superheating of liquid on the metal surface with respect to the saturation temperature and presence of surfaces irregularities on the metal which serve as starting points.

The mechanism of heat transfer is based on molecular transport of mass and heat by the vapour bubbles from the metal/liquid boundary layer. The vapour formation process and the duration affect the heat transfer process in boiling. Thus the interpretation of the boiling phenomenon lies in the understanding of the vapourization process of the quench medium.

Schwalm and Tensi (1981) observed that vapourization characteristics affect the surface reaction at the solid/liquid boundary in varying manner and consequently the transmitted heat flux. The "time-pattern" of surface reactions that occur during quenching operations can be classified into three (Moreaux and Beck, 1981). Schwalm and Tensi (1981) also differentiated three classes which they schematically represented as shown in figure 2.2.

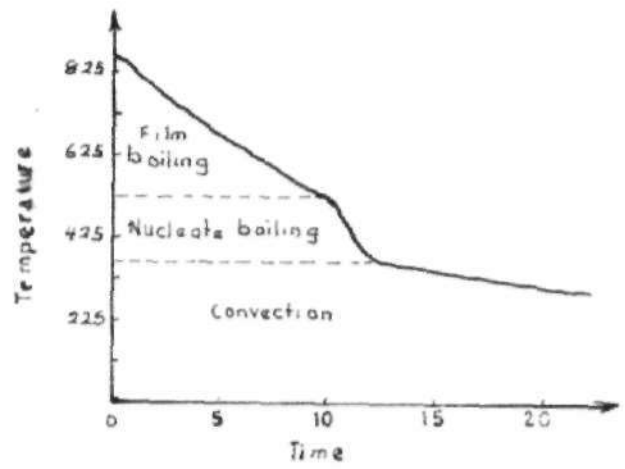
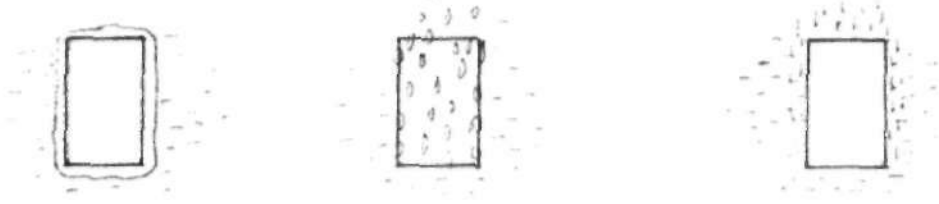


Fig 2.2 Schematic Temperature-Time Curve During Immersion Quenching of a Metal Cylinder



Regime I
Film Boiling

Regime II
Nucleate Boiling

Regime III
Natural Convection

Fig 2.3 Surface Reaction During Immersion Quenching

(a) Regime I -- Film Boiling:

When the metal piece, which has previously heated to a high temperature of between 850 ^{o}C to 1200 ^{o}C, is immersed in the liquid, the sample is blanked by a film of liquid vapour. The liquid is then separated from the heated surface by a layer of vapour and the heat flow reaching the liquid-vapour interface passes through a layer of vapour of low thermal

conductivity. Because of the insulating effect of the film, heat transfer h, is low; about 5.68×10^{-2} to 4.99×10^{-2} $W/(cm)^{2 \cdot o}C$ (Stolz et al, 1959). The heat reaching the liquid is, in turn, distributed by natural convection. With an increase in surface temperature, radiant energy transfer comes into play, complimenting that by conduction. Welty, et al (1976) reported that this occurs when the temperature difference (T_o - T_s), in the case of water at 100 ^oC, is above 300 ^oC (approximately).

For a saturated liquid, the heat flux from the heating surface at 540 ^oC and above, according to Beck (1981) is absorbed in vapourization of the layers of liquid at the vapour film border with a fraction being expended on super heating the vapour film.

In a sub cooled liquid, the heat passing through the vapour from the boiling surface is partly transferred further into the bulk of the liquid by convection. The rate of transfer is dependent on the degree of sub-cooling and rate of forced circulation. The two factors favourably influence the process of heat transfer (Isancheko, et al 1974).

Soltz et al (1959), in their work with paraffinic oils reported that the film boiling stage occurred within the temperature range of 870 ^oC to 490 ^oC.

(b) Regime II -- Nucleate Boiling:

As the temperature of the metal decreases, boiling is characterized by the rapid formation of bubbles promoted by presence of "nuclei" such as surface irregularities, impurities in the liquid and the superheating of the liquid.

Stolz et al, (1959) found that this stage of boiling started at 443 ^{o}C for the oils they tested. Moreaux and Beck (1981) observed the onset of the phenomenon of nucleation boiling at a temperature of 525 ^{o}C. They stated that the occurrence was observed when the difference in metal/fluid temperature was within the range of 130 ^{o}C to 200 ^{o}C.

The continual existence and growth of the vapour result in surface boiling changing into the boiling of the bulk liquid. This occurs when the liquid reaches saturation temperature due to superheat which is maximum directly at the heated surface.

The bubble grows only when the vapour pressure inside the bubble is less than the sum of all the forces acting upon it; that is, pressure exerted by the surrounding liquid and the surface tension of the bubble itself.

Welty et al, (1976) reported that the intensity of vapourization on the surface depends on the degree of superheat of the liquid which generates the necessary pressure. The temperature and pressure difference are given as follows:

$$T = \Delta P \, (dT/dp) \qquad \text{.....(2.14)}$$

$$P = \Delta P_1 + \Delta P_1 \qquad \text{.....(2.15)}$$

$$= \frac{2\sigma}{R_b} - \frac{\rho_L}{\rho_L - \rho_L} \qquad \text{.....(2.16)}$$

In nucleate boiling the main factor of heat is transferred from the heating surface to the liquid phase because the conductivity of the liquid is much greater than

that of the vapour. This occurs at P=1bar, K_{water} = 0.68 and $K_{(steam)}$ = 0.02 W/m ^{o}C (Grober and Erk, 1961). The vapour bubbles influence this process hydrodynamically by virtue of their stirring action. This action brings fluid to constant contact with the metal and leads to a considerable increase in the intensity of heat transfer and a greater equalization of the temperature of the main body of the fluid. The heat supplied to the bubble, however, is drawn predominantly from the fluid.

Increase in the heat flux increases the excess surface temperature (T_o-T_s) which results in higher superheating. Since superheating determines the intensity of vapourization, bubble formation sites therefore increase (Grober and Erk, 1961). However, the frequency of separation of vapour bubble is independent of the heat flux and hence of the temperature excess (T_o - T_s) of the heating surface (Schwalm and Tensi, 1981). It depends on the critical volume of the vapour and hence on the properties of the liquid and on the heating surface (Isanchenko et al, 1974).

(c) Regime III -- Natural Convection:

When the metal temperature falls to a point which is too low to give rise to vapourization, the mode of heat transfer changes to that by convection. Transfer of heat by convection, usually turbulent at the beginning, ends in laminar flow (Welty et al, 1976). They also reported that as long as the heat flux q is less than 1.735 x 10^{-2} KW/cm^2 the heat transfer mode will be by free convection with a

turbulent boundary layer which can be described by the equation of the form

$$N_{NU} = C\ (N_{GR} \times N_{PR})^{1/3} \qquad \text{.....(2.17)}$$

Nucleate boiling, in general, is the most efficient stage of boiling in heat transfer and consequently in quenching. This region is bounded by the film boiling at the beginning and natural convection at the end. The temperature of transition between this three stages (from film boiling to nucleate boiling and from nucleate boiling to natural convection) will determine the duration of existence of nucleate boiling and consequently the effectiveness of a quenching medium.

2.3 Assessment of Quenchants

2.3.1 Quench Severity

Cooling media may be classified as a function of their severity of quench or drasticity and may be represented by the time necessary to cool the metal from a given high temperature to room temperature or preferably 300 ^{o}C (Beck, 1961).

For steel, quenching has two desirable effects on the solid.

(i) a structural effect

Fast cooling at high temperatures prevents structural transformation leading to the equilibrium structure and makes possible martensitic transformation.

(ii) a mechanical effect

Fast cooling at low temperature may be undesirable because it introduces mechanical stresses between the superficial and the deep zones of the solid.

Hence, the most suitable quenchant is that which provides a more severe quench (faster cooling) at high temperature and slow at low temperature.

A test for evaluating quench severity, based on modification of the quenching technique developed by Grossmann et al (1930), was developed by a division of General Motor Company, Detroit Diesel. In the Grossmann procedure, end quench test results were correlated with those from immersion quenching of cylindrical bars of varying diameters. Equivalent diameter that has similar centre hardness to that of a particular end quench distance was then obtained for a specified quench. By reversing the procedure, Detroit Diesel employed a two diameter bar to evaluate quench severity. The bar was quenched under production condition and centre hardness values were taken at mid-length of both diameters. The values were plotted on the Grossmann Chart and used to define lines which give the quench severity of the different media.

2.3.2 Analysis of Properties of Quenching Media

The most important parameters in quenching operation are the stages or time periods (in relation to temperature) of manifestation of the different phases of boiling. These stages depend on the vapourization characteristics of the

medium and these in turn on the chemical and physical properties.

The variables which affect the heat extraction ability of the liquid include flash point, viscosity, stability of oils, oxidation resistance, thermal conductivity, specific heat, temperature of bath and immersion depth of sample, amongst others, as assessed by Paschkis (1958).

The oils under investigation are "Local Oils" which are essentially fatty oils. Property analysis are however, based on established parameters as used for known quenching mineral oils and solution of organic polymers.

(a) Chemical Analysis:

Fatty oils consist predominantly of fatty acids in glycerine molecules. They are polar substances having very long carbon chains with one or more chemically active/polar groups at one end. The properties of these oils are determined by their fatty acid radicals which may contain some reactive unsaturated and unreactive (saturated) acids.

Evans (1948) reported that the relative amount of these polar substances in fatty oils has been found to be a measure of degree of unsaturated fatty acids in the oil. Hence, the iodine that is absorbed by 100 grams of oil gives a numerical value indicating the amount of reactive or polar radical in the oil.

Naylor (1967) reported the presence of chemicals such as sulphur, phosphorus or chlorine in the oils under investigation. These elements react chemically with metal surfaces under heat by forming metallic soaps.

Chemical analysis of the oils being investigated was conducted by Ajala (1987). He determined the chemical composition of the oils and evaluated the saponification values, percentage free acid value, iodine value and percentage phosphorous content of each of the local oils (Table 2.1).

Table 2.1 Free Acid Value, Saponification Value Iodine Value And Phosphours Content Of Each of the local oils Investigated:

Oil	% Free Acid	Saponification	Iodine Value	Phophorus Content
Palm Oil	6.9 - 31.9	225.4-227.6	64.5	0.05
G/Nut Oil	0.1 - 0.6	223.0-230.2	106.0	0.07
S/butter oil	14.7 - 52.9	229.3-236.2	99.7	0.06

McGannon (1971) defines some of these properties as follows:

Saponification value - Indicates the ease by which the oil can be degreased effectively in an alkaline medium.

Iodine value - is a measure of the level of unsaturated fatty acid and it gives the amount of reactive or polar radicals in each of the local oils

Stability - This is determined by the percentage free acid value.

Oxidation Resistance - Oxidation results because of exposure of some oils for some period of time. It causes the rapid thickening of the oils due to the presence of suspended particles of the oxidation products and eventually a sludge is precipitated.

Pour Point - It is the lowest temperature at which an oil will pour or flow when it is chilled without disturbance, at a fixed rate.

Flash Point - The temperature at which a flash (momentary ignition) occurs at any point on the surface of an oil when a flame is passed over it but the oil does not continue to burn.

Fire Point - The temperature at which ignition occurs at any point on the surface of an oil when a flame is passed over it and the oil continues to burn.

Neutralization Number - Is the number of milligrams of potassium hydroxide necessary to neutralize the acid content of one gram of oil under standard test condition.

Demulsibility - The ability or the extent to which water separates out of an oil emulsion. The presence of oxidation products may retard or even prevent the separation process.

Viscosity - Is defined as the ability of the oil to resist flow or shear.

2.4 Quenching Practices

2.4.1 General

The major consideration in quenching is hardenability as determined by the carbon and alloy contents in the steel. However, the quality of treatment depends largely on the quenching technique and the characteristic of the medium. The ability of these techniques to achieve variability in quenching rates or improved heat transfer properties is based mainly on the heat transfer rate (h) of each medium

Quenching heat treatment has relied mainly upon immersion quenching. Accordingly, the heat transfer rate h at the surface of the metal determines the rate of cooling throughout a part. On the other hand, the stability and duration of the film boiling phenomenon which occurs during immersion quenching affects the heat transfer rate at the metal surface (Jones, 1974) Immersion quenching is usually carried out in water and mineral oil though other quenching media such as liquid nitrogen, water sprays, salt brine, and polyvinyl alcohol have been used on an experimental basis (Steve, 1951).

Quenching in water, though inexpensive, sometimes causes severe distortion and cracking. Moreaux and Beck (1981) observed that the most important parameter as regards quench characteristic is the temperature. While flexibility in film boiling and stability can be obtained by fluid temperature control, the full advantages of the high heat extracting capabilities can not be obtained because the process is only moderately successful (Croucher, 1971).

Stevens and Witte (1973) showed that film boiling is very stable during quenching in water at 100 ^{o}C and is impossible to distabilized, however this is not so in the case of sub-cooled water. Explosive and random distabilization of film boiling observed in the latter case leads to unreproducibility of cooling curves.

Essentially, stability of the film boiling grows both with the initial temperature of the metal T_O, and T_S, the temperature of the surrounding. However, it grows more

rapidly with T_s than with T_o (Moreaux et al, 1981). The most important parameter being the temperature gradient in the liquid to the sub-cooling (100 ^oC - Ts). They used this to show the stability of film boiling at the intermediate temperature range of Ts = 60 ^oC, but only for when T_o is greater than 680 ^oC.

In oil quenching, quench cracking observed during water quenching is minimized as the cooling rate is much slower (Steve, 1951)

The chemical compositions of the oils determine their vapourization characteristics which establishes their cooling power. Besides these, the viscosity indices of the oils also play a major role (Naylor, 1967).

In general, oil quenchants fall into four categories (Weymueller, 1972). These are conventional, fast oil quenching, hot oil quenching, and marquenching.

The conventional quenching oil are essentially mineral oils with viscosity range of 70 to 300 SUS at 38 ^oC. Bath temperature is usually 49-66 ^oC though higher bath temperatures are used when slower cooling rates are desired during the convention stage of cooling. High-carbon and high alloy steels (AISI 4140, 52100 and 6150), having low critical rates are usually quenched in these oils. Stresses are minimized due to slow cooling while desired hardness is also realized.

Fast quenching oil normally has low viscosity 70 - 125 SUS at 38 ^oC. Bath temperature is usually 49-66 ^oC. Inhibited mineral oils are used because they exhibit

excellent thermal and oxidation stability while promoting maximum heat transfer in the first two stages of cooling (Service, 1947). The oils are used for low carbon and lean alloy steels (AISI 1010 and 1020).

Hot quenching oils include those with viscosity in the range of 250 to 3000 SUS at 38 ^{o}C. These are either inhibited or straight mineral oils and used between 93 and 149 ^{o}C. The oils generally minimize distortion and cracking in parts with intricate contours. Greater tensile and fatigue strengths can be attained in thin - wall parts of alloy steels such as AISI 8620 in hot quenching oils.

Marquenching oils usually have viscosity of 2000 SUS at 38 ^{o}C. This oils are used at bath temperature of 149 ^{o}C for steels having low critical cooling rates and where absolute minimum cracking and distortion is desired. Large pieces are normally quenched in this manner to ensure similar core and surface hardness.

Beck (1981) in his work on three different oils, (light paraffinic, aromatic and naphttanic), showed that the transition temperature between film boiling to nucleate boiling, and nucleate boiling to convection, are mainly determined by the viscosity of the oil. There was, however, little variation of the duration of film boiling with temperature of the bath, and thus with its viscosity. The study showed the dependence of film boiling on the nature of the oil which can be characterized by the intrinsic viscosity. Effect of high oil temperature mainly serves to

speed up equalization of oil/metal temperature thereby slowing down cooling and minimizing distortion in the piece.

Agitation helps in destroying surrounding vapour barrier on the metal. The major effect is recorded during the vapour and boiling phases (Steve, 1951).

Once the metal temperature drops below the boiling range, the oil constantly contacts the work and the effectiveness of agitation rapidly decreases. This same effect is found in the case of water (Steve, 1951).

Stolz et al, (1959) found that shallow immersion of less than about one sample diameter yielded slower cooling than at greater depths. They explained this occurrence on the basis of proximity of oil/air interface which constitutes a boundary similar to a solid wall and tends to restrict the circulation of the quenchant.

Moreaux and Beck (1981) explained that upon contact of the hot metal with the oil, the oil undergoes pyrolysis which leads to formation of stable spices (aromatic and poly aromatic hydrocarbons). This ageing, they say, gives rise to a reduction in the duration of film boiling and a lowering of the starting temperature of natural convection of a pure mineral oil.

In essence, Evoucher (1971) reported that oil quenching serves to reduce quench rate and quench cracking tendencies, though it may cause severe fire, smoke and pollution problems.

If, however, the bath temperature is about 66 ^{o}C less than the oil flash point, then the risk of fire is reduced during quenching (Naylor, 1967). Naylor recommended that, to ensure adequate safety, oil with high viscosity (2,000 SUS) should be employed especially in hot-oil systems since higher flash point margins will be realized.

2.4.1 Simulative Hardneability Tests

The problem of hardenability depends on how several parameters influence (enhance) the formation of martensite.

Siebert et al (1977) reported that the first standard test for hardenability was published by Shepard in 1930 when standardization of the quench and size of the test pieces of steel was suggested. Fracture appearance was later examined as a means of assessing the depth of hardening. The test was designated "Shepard P-F test", where P stands for penetration or depth of hardening and F indicates fracture appearance.

Several other methods were proposed. One still in use as a sensitivity test for shallow hardening steels was proposed in 1938 by Burus, Moore and Archer. It is called the "SAC Test" (Houghton, 1960).

The S.A.C test involves quenching of 25.4 mm diameter steel bar in water and determining hardness traverse across the diameter. Hardness value in Rockwell C (Rc) at various depths below the surface are then plotted to report the surface hardness S, the area under the curve, A, and the centre hardness C, from which the test derives its name. It

offered a means of comparing the hardenability of different steels.

Jominy end quench test, which achieved wide industrial application, was proposed by Jominy and Boegehold (Bain and Paxton, 1966). The original idea was for carburizing steels. It has however undergone several modifications including usage for non-carburizing steels. The test, however, has limited application because it is only suitable for steels with "medium" hardenability (Bain and Paxton 1966).

The first approach to placing all steels on a common basis for hardenability purposes was first published by Grossmann, Asimow and Uban in 1939 (Siebert et al 1977). The method combines the effects of material and media parameters. The test involves the use of various cylindrical specimen of varying diameters quenched in various media of different severity of quench.

Through this method, the "critical diameter" D, for each quench is defined as the diameter that will just harden throughout to a structure of 50 percent martensite (and 50% transformation product) at the centre. The centre hardness is then plotted against diameter to obtain the hardness penetration.

Assessment of the available hardenability tests separately does not generally lead to a conclusive judgement (Siebert et al, 1977). To overcome this problem, Grossmann et al (1930) and then Lamont (1943) correlated data for both tests (the end quench test and cylindrical bar tests) based on equal cooling times. This same technique is being adopted in this research work.

CHAPTER III

3.0 EXPERIMENTAL PROCEDURE

3.1 Equipment

The main equipment used for this research work was an electric induction furnace capable of attaining 1000 oC and equipped with thermostat for regulation purposes.

A vickers hardness testing machine was used for hardness measurements and metal containers of sufficient volumetric capacity were adapted as baths for quenching.

3.2 Specimen Preparation

Test specimens were machined from a 32mm diameter steel rod obtained from Katsina Steel Rolling Company. All samples were of the same heat number and of the following composition:

%C	%Mn	%Si	%P	%S
0.35	0.90	0.030	0.04	0.04

Steel grade designation, St 60-2, is based on DIN 488 standard. The composition conforms to British standard BST 47/50 RU and Society of Automotive Engineers (SAE) grade 1035.

Test samples were of cylindrical profile and machined finished. Prior to austenitizing, the samples were normalized at 900 oC. To reduce decarburization to the bearest minimum during heating, test samples were put in small boxes filled with cast iron chips. The boxes were then covered and sealed with clay.

3.3 Grossmann Test Procedure

Grossmann test procedure was employed for quenching and adapted to evaluate quench characteristics of media under study.

Cylindrical bars of five different diameters (27.90mm, 25.40mm, 19.05mm, 12.70mm and 6.35mm) were quenched in each of the media. The dimension of each of the sample was such that the length was five times its diameter resulting in a large "surface to volume" ratio. The ratio approximates an infinite length dimension, thereby eliminating possible end cooling effect (Moreaux and Beck, 1981). The cooling at the centre of each piece is therefore independent of heat loss from the ends.

Following laid down procedures (Lesile, 1982), samples were austenitized at 875 oC and left to soak at that temperature for 30 minutes before being quenched. After quenching, each cylinder was sectioned at the middle and test pieces taken from each half for metallography and hardness measurements.

Temperature of each oil investigated at the time of immersing the steel was at the flow point. However, "reference experiments" were conducted by raising the temperature of each of the oils to, at least, two and a half times the flow temperature.

This enables an appraisal of the effect of bath temperature.

Test conditions were as given in the table 4.1

Table 3.1 Test Condition

SPECIMEN SIZE (mm)	LIQUID VOLUME (LTS)	BATH TEMPERATURE (^{O}C) PALM OIL	G/NUT OIL	S/BUTTER OIL	COLD WATER	HOT WATER
27.90	28	28	26	29	26	80
25.40	24	"	"	"	"	"
19.05	18	"	"	"	"	"
12.70	12	"	"	"	"	"
6.35	6	"	"	"	"	"
25.40(REF Quench Severity Specimen	24	75	65	80	--	--
	32	28	26	29	26	80

3.4 Jominy End Quench Test:

Jominy End Quench Test method was employed to evaluate the hardening characteristics of the steel. The test piece was a 25.4mm diameter steel cylindrical specimen machined out of the 32mm diameter steel rod used for the Grossmann test specimens.

Specimen was austenitized at 875 oC and subjected to end cooling by a jet of water from an orifice. Water pressure was such that a 64mm free jet height is maintained.

After the austenitized specimen was dropped into the holding fixture, the pump was switched on and stream of water impinged upon the test bar until the bar cooled to room temperature. Hardness measurements were taken at 1.6mm interval from the base of the piece along a 0.38mm deep flat ground longitudinally on the bar.

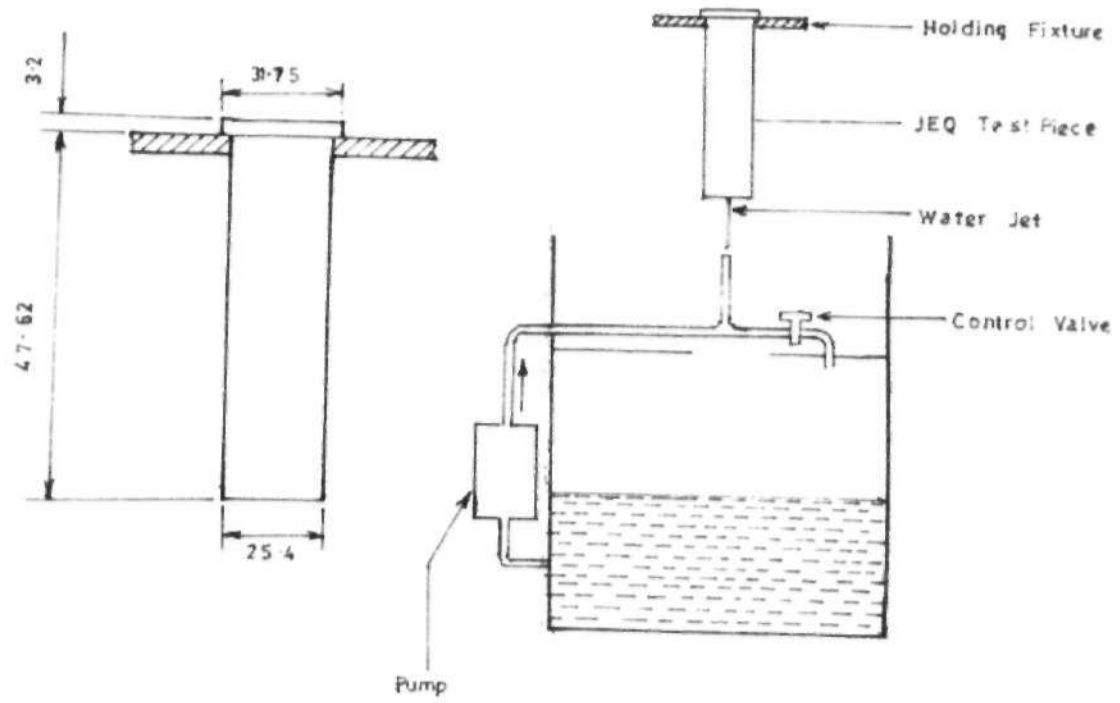


Fig 3.1 Jominy End Quench Test Apparatus

3.5 Quench Severity Test

The test was based on the procedure developed by a division of General Motor Company. Test piece machined from the same steel sample was made up of two different diameters; (26.4mm and 12.7mm), as shown in figure 3.2. This was quenched after heating to 875 oC. Hardness measurements were then taken at the centre of each diameter after cutting at mid length positions. The values obtained were used for plots on the modified Grossmann chart to obtain the quench severity of each oil under investigation.

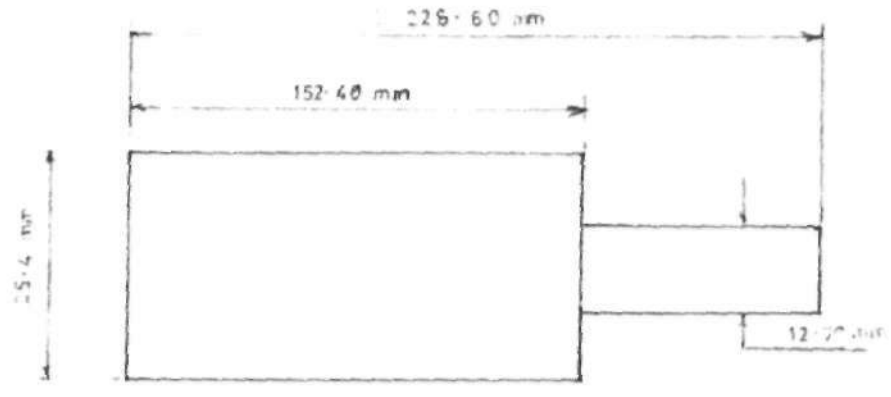


Fig 3.2 Quench Severity Specimen

3.6 Quench Media

A total of five media comprising of palm oil, ground nut oil, shea butter oil, cold water and hot water were investigated.

The oils were obtained from Zaria and used as purchased. To eliminate possible effect of oil decomposition on quench characteristics, the oils were changed after each quenching operation. Bath volume was made out to exceed the minimum "bath to specimen" volume ratio (Vl/Vs) of 1 liter bath volume to 3446 mm^3 metal volume as recommended by Moreaux and Beck (1981). It was also ensured that the sample was adequately submerged in the quenchant with more than 10mm of the liquid remaining above its upper face.

3.6.1 Viscosity

Since the quench characteristics of an oil depends on the intrinsic viscosity of the oil (Moreaux, 1981), the viscosity of the oils were thus determined in accordance to the procedure stipulated by the Institute of Petroleum

(Evans, 1984). It was determined at room temperature of 26 ^{o}C and at 60 ^{o}C using the Redwood Viscometer. The numerical value represents the number of seconds required for 50 milliliters of the oil to efflux through a standard orifice under a standard falling height at a particular temperature. The value is expressed in Redwood Seconds.

3.7 Parameter Assessment

Hardness measurement and metallographic examination of the quenched specimens were used to assess the effect of the oils under investigation. Samples were thoroughly cleaned with fine abrasive paper after quenching before the assessment tests were commenced.

3.7.1 Hardness Measurement

Vickers hardness testing machine was used to determine the hardness across the traverse section of the mid length position of the cylindrical specimens used in Grossmann test. (Figure 3.3)

Specimens were initially ground and finished on fine abrasive paper No.000 before hardness measurements were taken along all four orthogonal radii on the traverse surface. Positions of hardness measurements on each radius were at the centre, 1/4 - radius, 1/2 - radius and 3/4 - radius.

For the end quench test, a 0.38mm deep flat surface was ground along the length of the specimen and hardness measurements were made at 1.6mm intervals from the quench end.

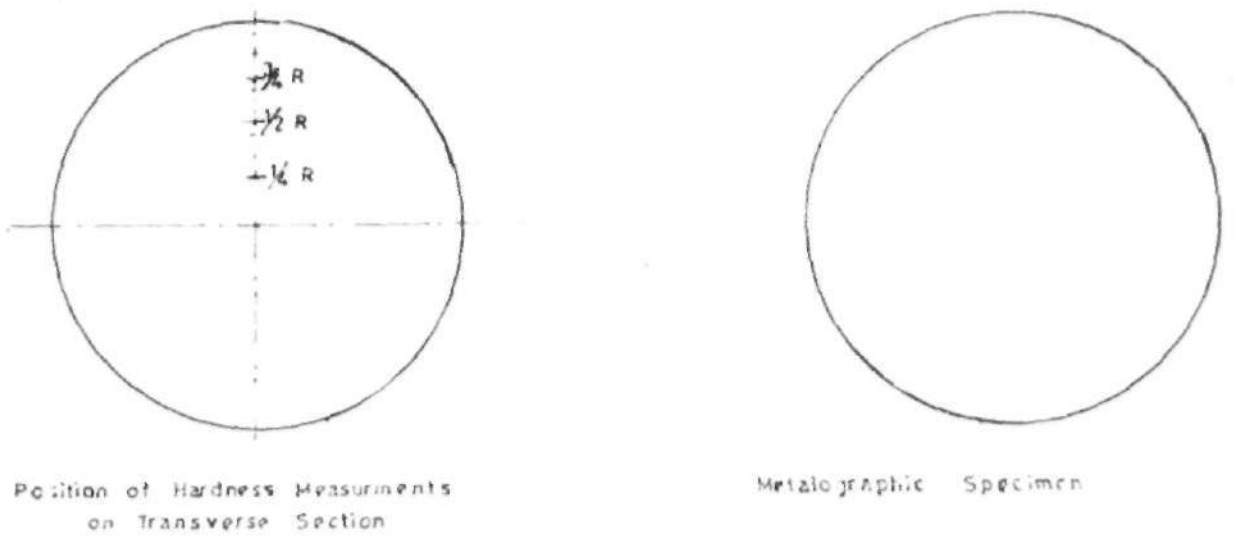


Fig 3.3 Grossmann Test Specimen (Parameter Assessment)

3.7.2 Metallography

(a) Macro-Examination:

The metallographic specimens were carefully ground on progressively finer grades of emery paper No.220 and No. 320. It was then etched in 50% hydrochloric acid at 70 ^{O}C for 15 minutes. The specimens were preheated to the etchant temperature in water prior to immersion. After etching, the specimen were thoroughly washed in water and finally in alcohol before being macro examined for depth of hardening.

(b) Micro-Examination:

Specimens used for macro-examination were subsequently subjected to further finer grinding on emery cloth No.400 and No.600 with proper rinsing to ensure temperature regulation of the piece while grinding. Polishing was carried out afterwards on a rotating cloth pad using aluminium oxide powder.

The specimens were then etched in 3% nital (Nitric acid and alcohol) by plunging into the reagent and agitating for 10 to 15 seconds. They were then withdrawn and washed in water and finally in alcohol to remove the last traces of water. The specimens were then micro-examined.Photographs of various samples at different magnifications were then taken.

CHAPTER IV

4.0 RESULTS

The results of Grossmann test in relation to average hardness values obtained on four perpendicular radii are given in table 4.1. Hardness traverse for various round bars quenched in media under investigation are shown in various plots

Micrographs of the traverse section of the middle part of the specimen are shown in various plates. The plates depict microstructures of the specimens after quenching in each medium. To evaluate the effect of both temperature, 25.4mm diameter specimens were quenched in each of the oils at two selected temperatures; charts and micrographs for these are also included.

Result of the Jominy End Quench test was used to assess hardenability characteristics of the steel by correlation with the Grossmann test results. An estimate of the severity of quench of each medium was made using the modified Grossmann procedure developed by Ford General motors. In addition, quench severity was derived based on the steepness of the plot of specimen centre hardness against diameter for each medium. This is to verify results obtained in the previous method.

Measured values of the viscosity of the oils at room temperature, (26 ^{O}C) and at 60 ^{O}C are also presented in a table.

4.1 Grossmann Test Results

4.1.1 Hardness Measurements

Values obtained are given in Table 4.1. These values represent hardness readings (D.P.N.) obtained at the Centre, 1/4 radius, 1/2 radius and 3/4 radius positions of each specimen. Figures 4.1, 4.2, 4.3, 4.4 and 4.5 show the hardness traverses for 27.9mm, 25.4mm, 19.05mm, 12.70mm and 6.75mm diameter specimens for each of the media under investigation.

Figures 4.6, 4.7, 4.8, 4.9, and 4.10 show the hardness traverses of each of the diameters quenched in various media.

4.1.2 Effect Of Bath Temperature On Hardness Traverse Using 25.4mm Diameter Specimens

Results of hardness measurments carried out on the specimens quenched in the oils at higher bath temperatures are given in table 4.2.

Figure 4.11, 4.12, 4.13, and 4.14 represent the plots of the hardness traverse obtained for palm-oil groundnut-oil shea butter-oil and water respectively at diferent bath temperatures.

Table 4.1 Hardness Value Traverse of Quenched Steel Specimens

QUENCH MEDIUM	SPECIMEN SIZE	POSITTON OF HARDNESS MEASURMENTS ON SPECIMEN			
		CENTRE	0.25 R	0.5	0.75 R
PALM OIL	27.90 mm	182.00	182.00	179.00	185.25
	25.40 mm	182.00	181.75	184.75	200.25
	19.05 mm	216.00	215.25	225.75	264.75
	12.70 mm	227.00	227.25	237.50	239.25
	6.35 mm	412.00	446.00	465.00	-
GROUND - NUT OIL	27.90 mm	168.00	183.00	180.50	196.50
	25.40 mm	172.00	169.72	178.75	189.50
	19.05 mm	204.00	204.33	212.67	214.00
	12.70 mm	233.00	240.75	243.50	270.00
	6.35 mm	364.00	360.50	389.50	409.00
SHEA BUTTER OIL	27.90 mm	168.00	170.75	173.50	177.75
	25.40 mm	209.00	206.67	206.33	211.00
	19.05 mm	222.00	235.75	235.25	237.25
	12.70 mm	223.00	231.75	229.00	251.50
	6.35 mm	364.00	358.50	370.50	411.50
COLD WATER	27.90 mm	261.00	263.50	258.00	264.61
	25.40 mm	287.00	284.74	283.50	290.28
	19.05 mm	339.00	329.25	337.75	344.50
	12.70 mm	363.00	367.50	382.00	377.00
	6.35 mm	594.00	598.00	597.00	597.70
HOT WATER	27.90 mm	165.00	163.25	160.25	168.00
	25.40 mm	163.00	167.25	172.50	170.25
	19.05 mm	203.00	189.50	197.75	202.00
	12.70 mm	211.00	208.00	213.00	210.50
	6.35 mm	225.00	218.67	230.67	248.67

Table 4.2 Hardness Value Traverse of Steel Specimens Quenched in Baths at Elevated Temperatures

MEDIA	SPECIMEN SIZE(mm)	BATH TEMP.	POSITION OF HARDNESS MEASUREMENTS ON SPECIMEN			
			CENTRE	0.25R	0.5R	0.75 R
P-OIL	25.40	75	195.00	194.33	204.00	213.00
G/NUT OIL	25.40	65	176.00	177.67	180.33	194.33
S/BUTTER OIL	25.40	80	256.00	251.67	258.33	277.00
WATER	25.40	80	163.00	167.25	172.50	170.25

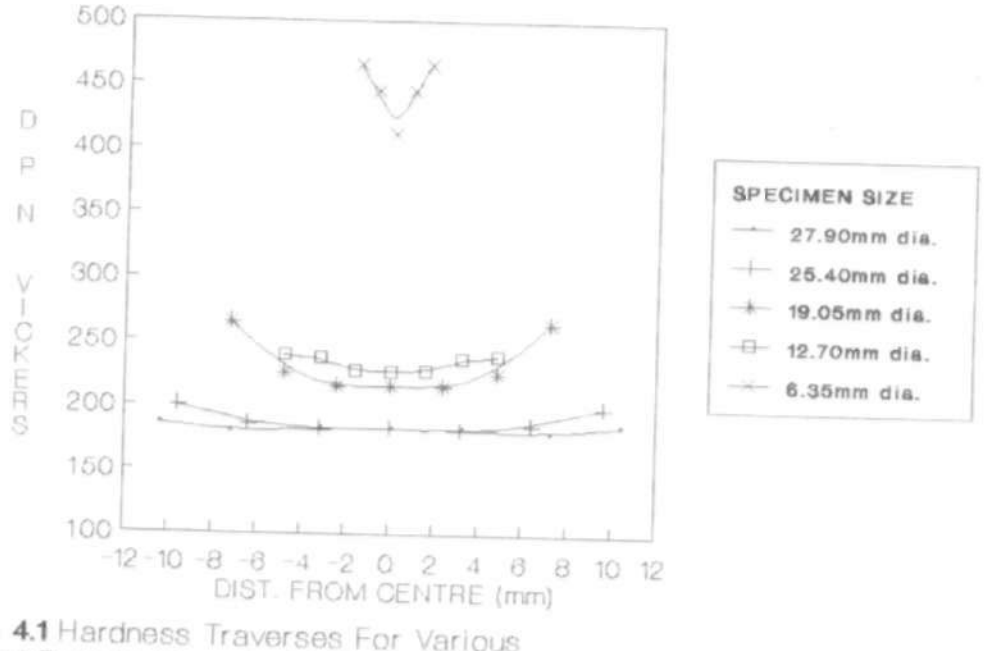


Fig 4.1 Hardness Traverses For Various Steel Rounds Quenched in Palm-Oil at 28 oC

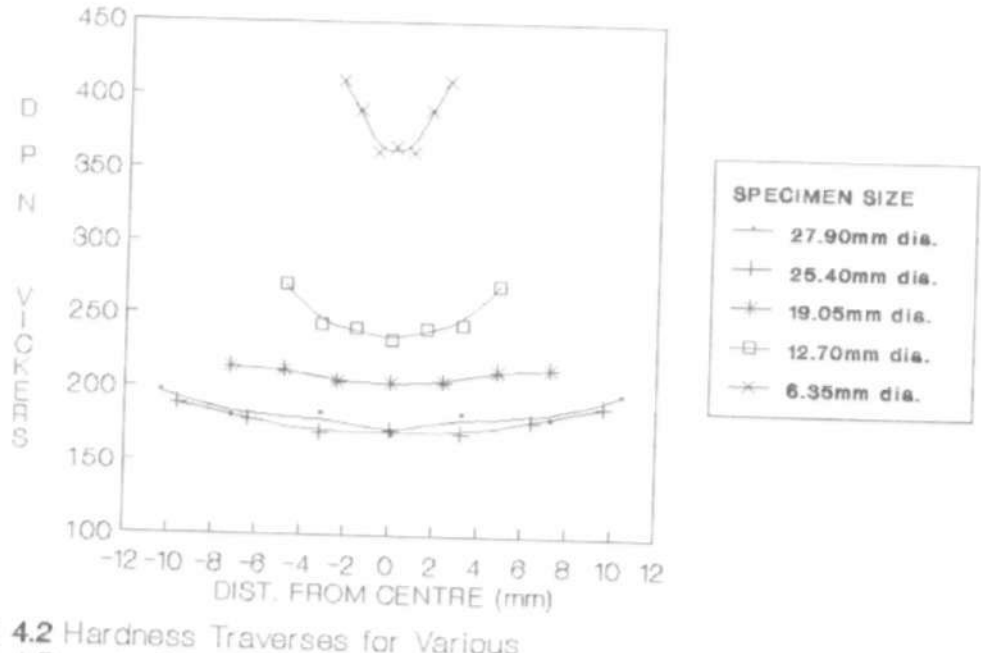


Fig 4.2 Hardness Traverses for Various Steel Rounds Quenched in G/nut-Oil at 26 oC

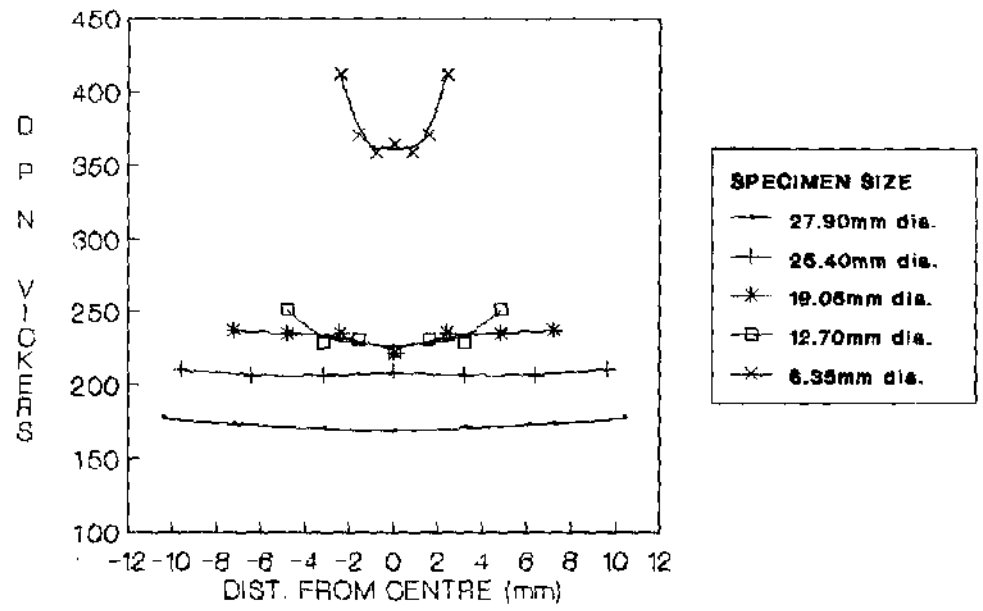


Fig 4.3 Hardness Traverses For Various Steel Rounds Quenched in S/butter-Oil at 29 oC

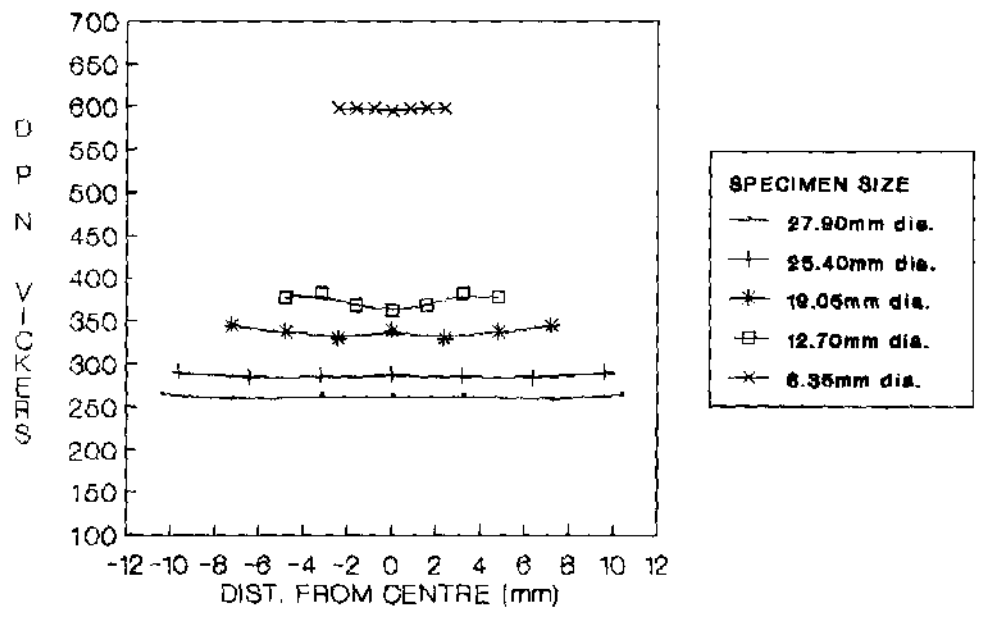


Fig 4.4 Hardness Traverses For Various Steel Rounds Quenched in Cold Water at 26 oC

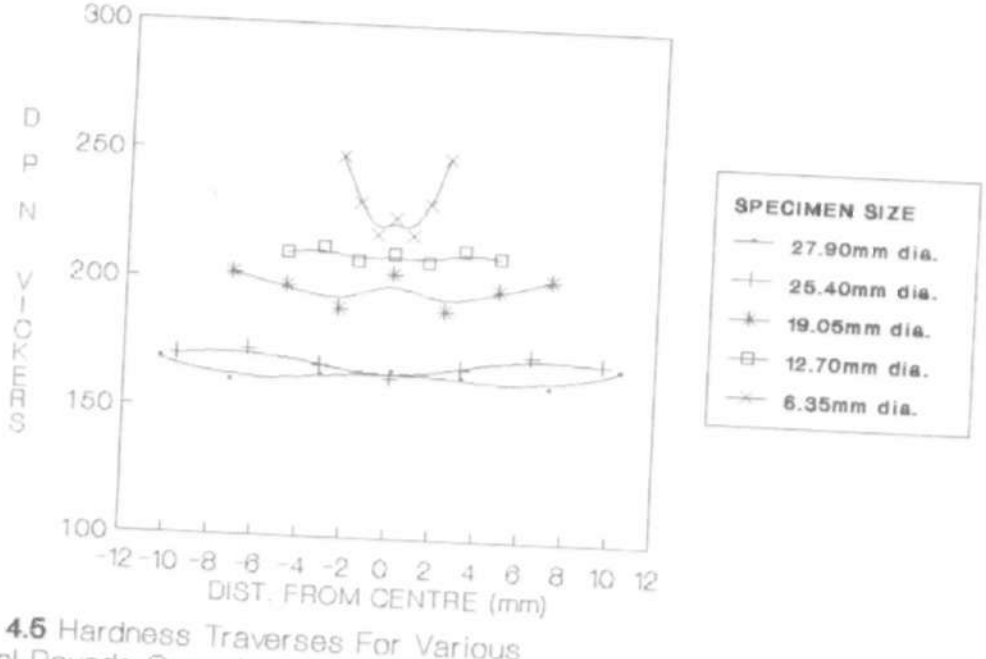


Fig 4.5 Hardness Traverses For Various Steel Rounds Quenched in Hot Water at 80 oC

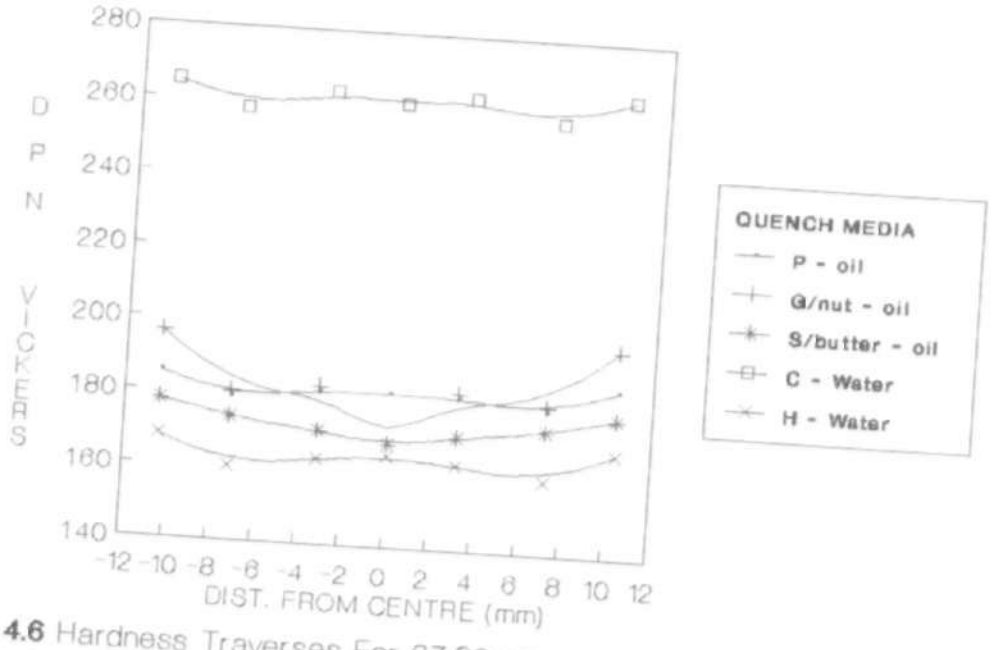


Fig 4.6 Hardness Traverses For 27.90mm Diameter Steel Rounds Quenched in Various Media.

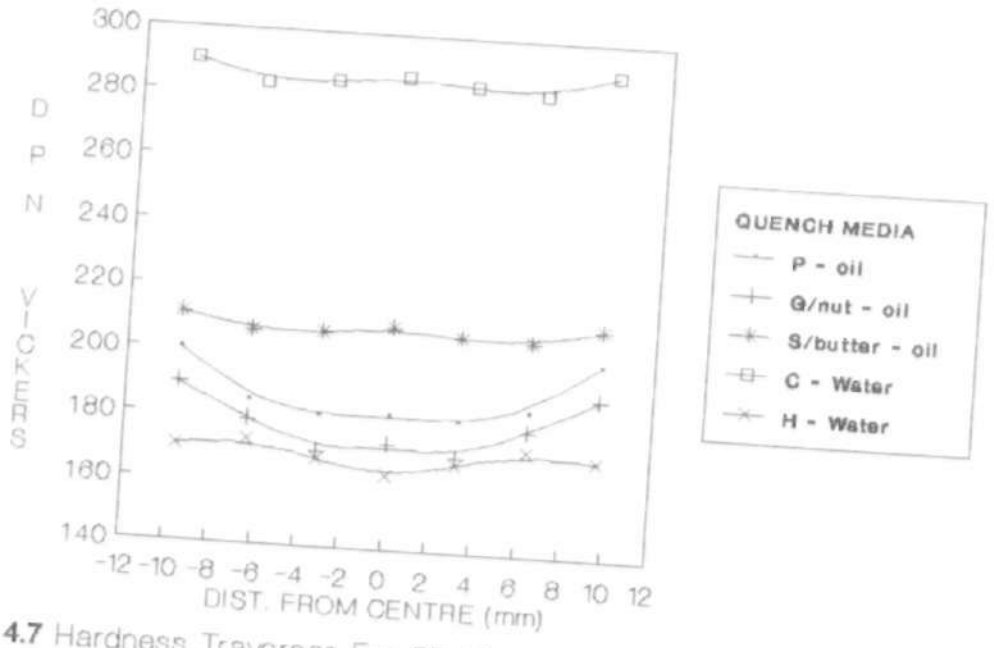


Fig 4.7 Hardness Traverses For 25.40mm Diameter Steel Rounds Quenched in Various Media

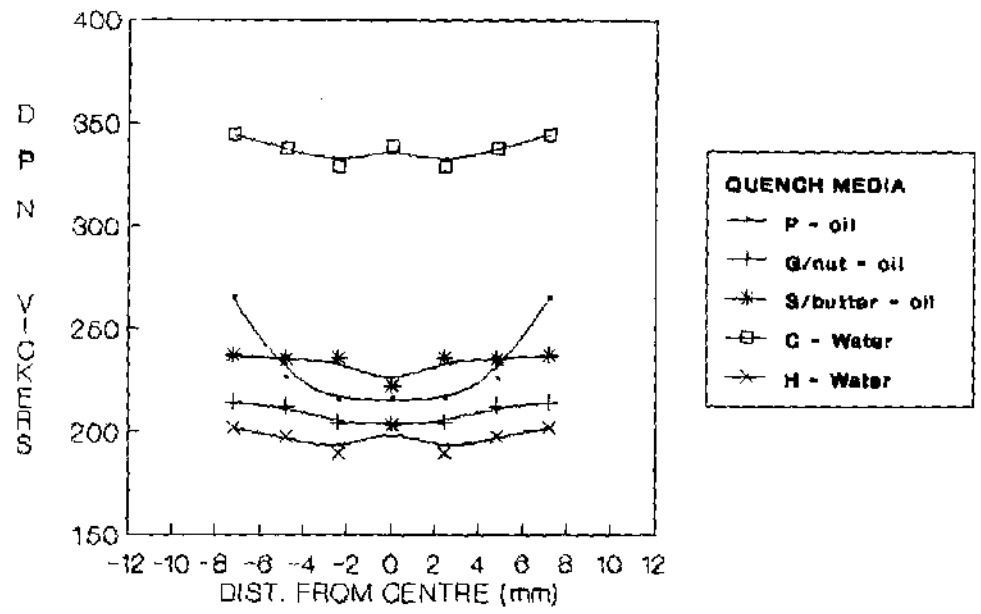


Fig 4.8 Hardness Traverses For 19.05mm Diameter Steel Rounds Quenched in Various Media

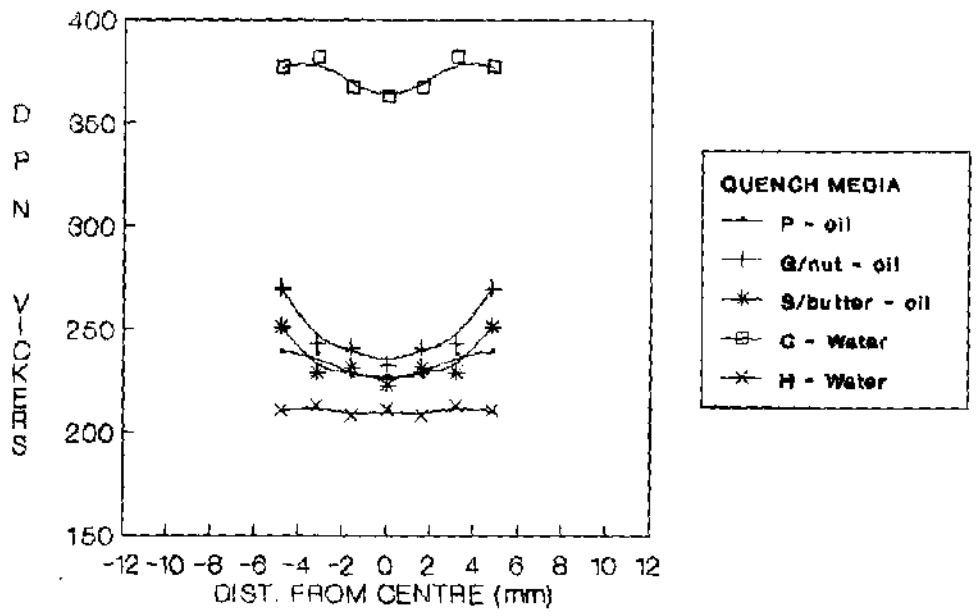


Fig 4.9 Hardness Traverses For 12.70mm Diameter Steel Rounds Quenched in Various Media

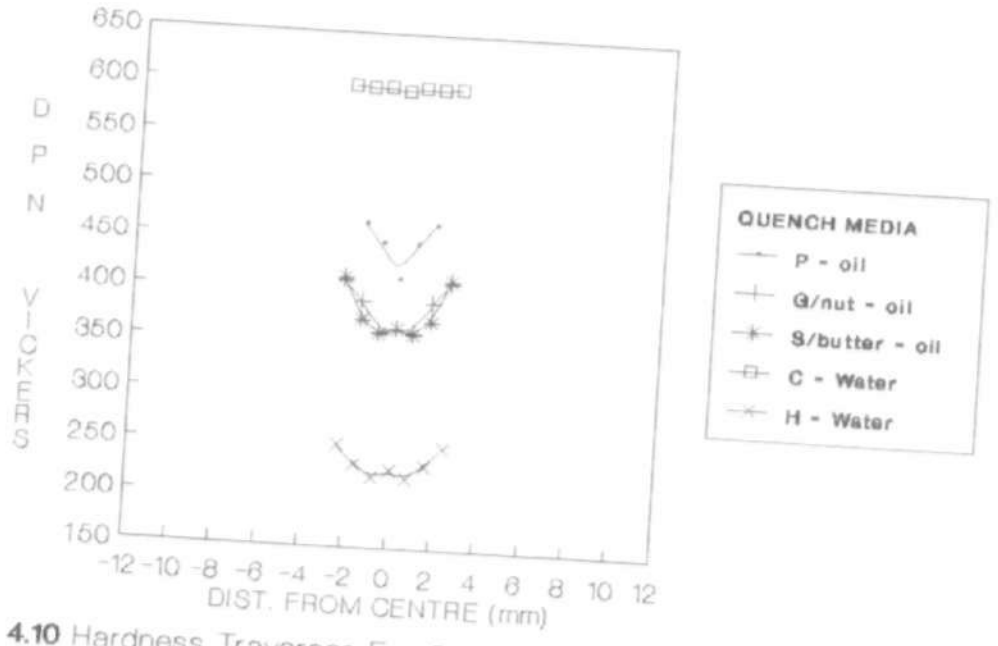


Fig 4.10 Hardness Traverses For 6.35mm Diameter Steel Rounds Quenched in Various Media

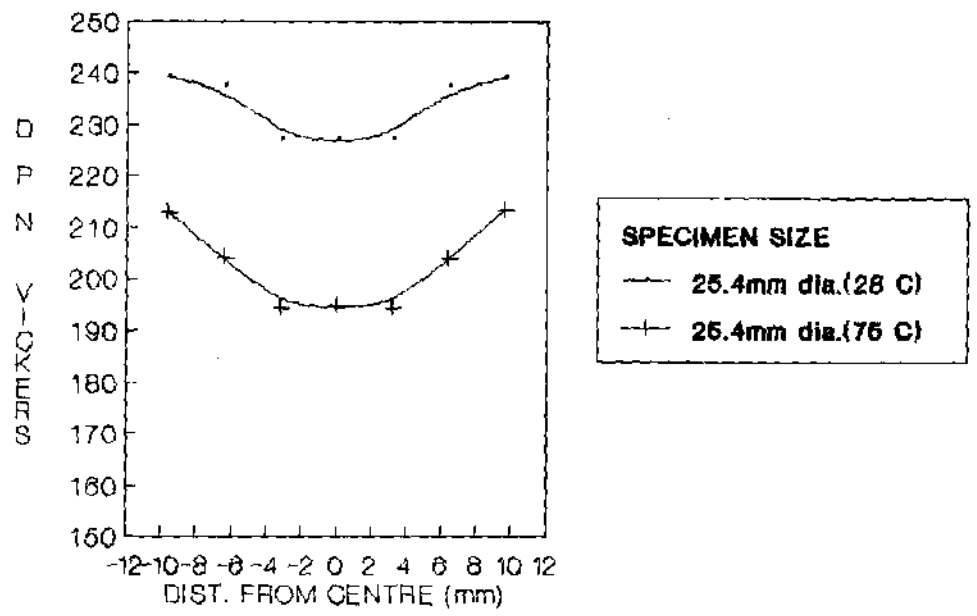


Fig 4.11 Hardness Traverses For 25.4mm Diameter Rounds Quenched in Palm-Oil at Different Temperatures

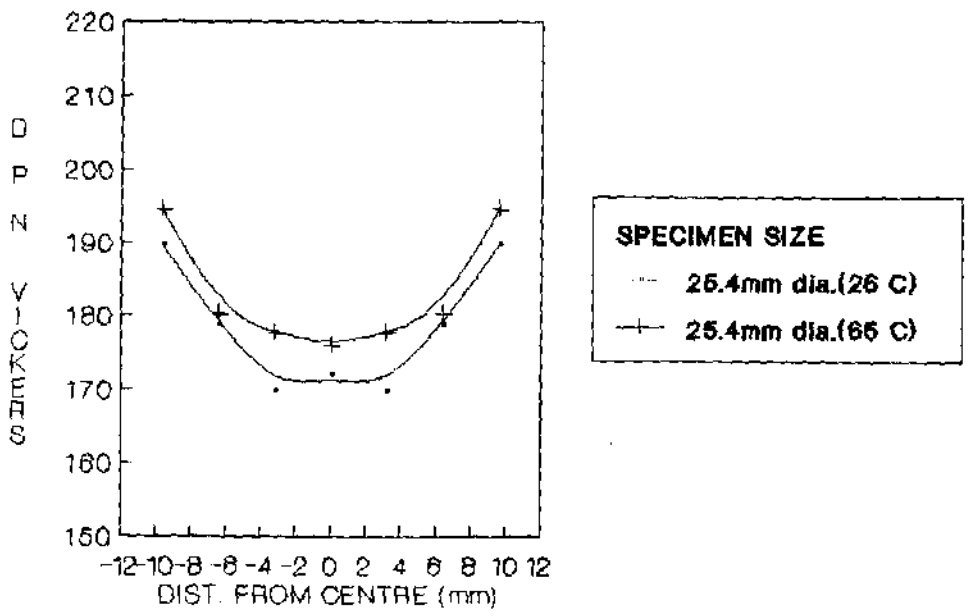


Fig 4.12 Hardness Traverses For 25.4mm Diameter Rounds Quenched in G/nut-Oil at Different Temperatures

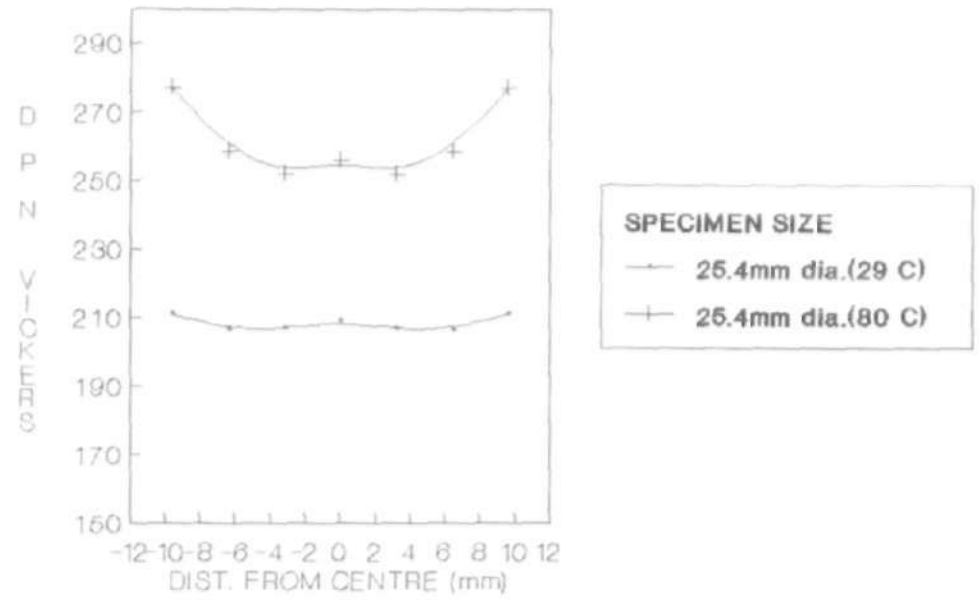


Fig 4.13 Hardness Traverses For 25.4mm Diameter Rounds Quenched in S/butter-Oil at Different Temperatures

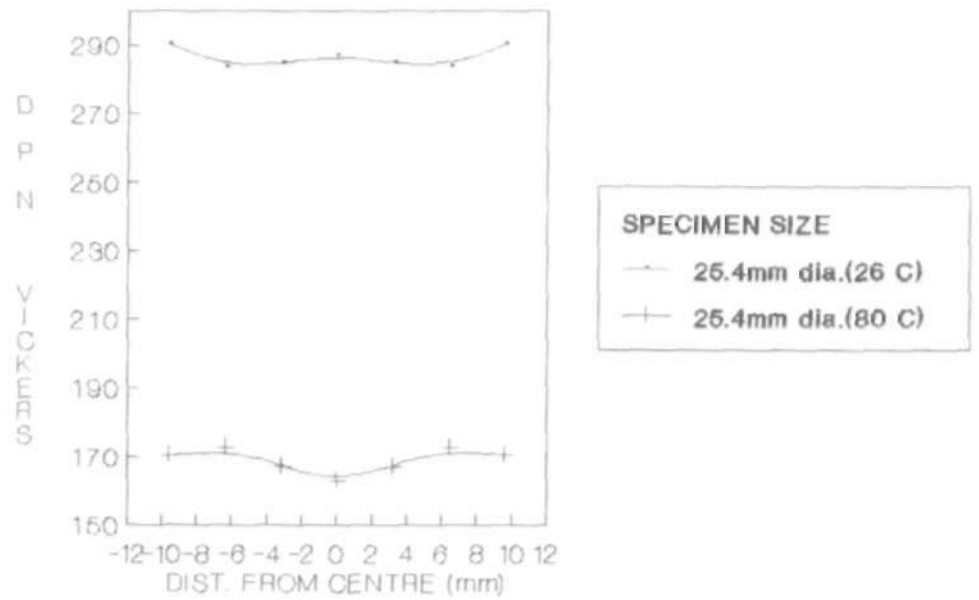


Fig 4.14 Hardness Traverses For 25.4mm Diameter Rounds Quenched in Water at Different Temperatures

4.1.3 Micrographs

The as rolled microstructure of the steel used was recorded at X200 and X500 magnifications (plates 1 and 2). These micrographs serve as references in the assessment of the microstructural changes which occurred as a result of the quenching operation.

Micrographs of each of the specimens quenched in the oils under investigation are given in plates 3 to 27. Plates 28 to 31 show the microstructures of 25.4mm diameter specimens quenched in the oils under study at the two different bath temperatures.

4.1.4 Micrographs of 25.4mm Diameter Steel Cylinder Specimens Quenched In Baths of Different Temperatures

The micrographs of specimens quenched in baths of higher temperatures are given in plates 28B, 29B, 30B, and 31B. These are for palm-oil, groundnut-oil, shea butter-oil and water respectively. Micrographs of similar specimens, quenched in baths of lower temperatures are presented alongside to facilitate comparison. These are shown in plates 28A, 29A, 30A and 31A.

4.2 Jominy End Quench Test Result

The Jominy End Quench test procedure was used in assessing the depth hardening characteristics of the steel. Figure 15 depicts a plot of hardness measurement (table 4.3) taken at 1.6mm interval along the length of the bar.

In correlating the Jominy end quench test results and the Grossmann test results lines representing centre hardness of quenched bar equal to particular hardness value on the Jominy end quench bar were super-imposed on the Jominy end quench test plot . This relates the mass of bar to the end-quench test. Figures 4.16 and 4.17, illustrate the plots for the oils and water respectively.

4.3 Quench Severity

Results of assessment of the quench severity of the oils investigated are illustrated in figures 4.18 and 4.19.

Figure 4.18 shows a plot of hardness values measured at the centre of each bar in progressively larger diameters. The cooling power of each medium is reflected in the steepness of the curve for each medium.

Rating of the oils by the method of Rushman (1963) enabled evaluation of the cooling power of individual quench bath. The hardness values obtained at the radii of the specimen are given in table 4.4.

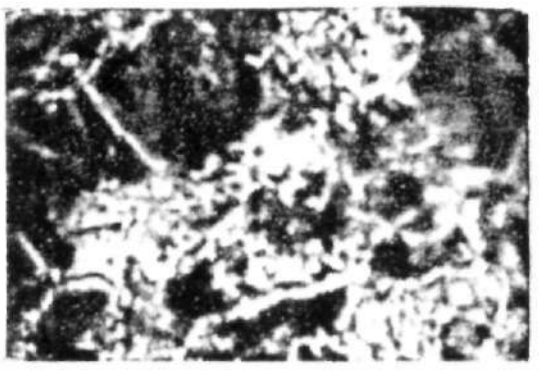


PLATE 1

MICROGRAPH OF 0.35% C STEEL (AS ROLLED)
X200, ETCHED IN 3% NITAL

Plate shows ferrite (white) structures and also outline of prior austenite grains. Black region is pearlite.



PLATE 2

MICROGRAPH OF 0.35% STEEL (AS ROLLED)
X500, ETCHED IN 3% NITAL.

Structure is same as in plate 1. The coarseness of the pearlite (black) structure is however better resolved.

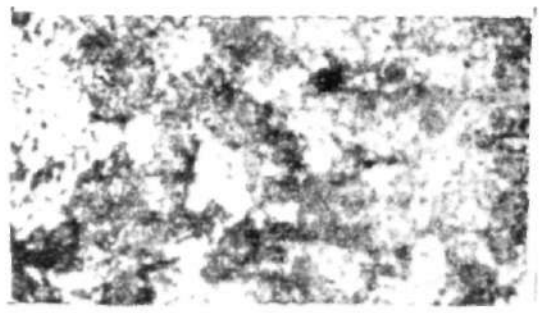


PLATE 3
P-OIL QUENCH

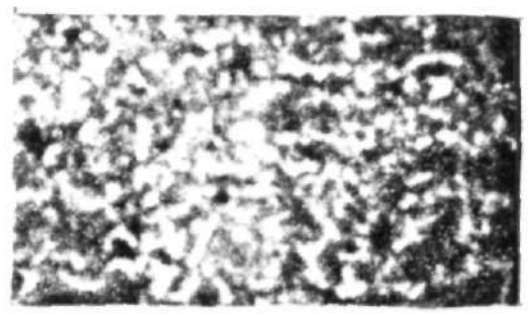


PLATE 4
G/NUT-OIL QUENCH

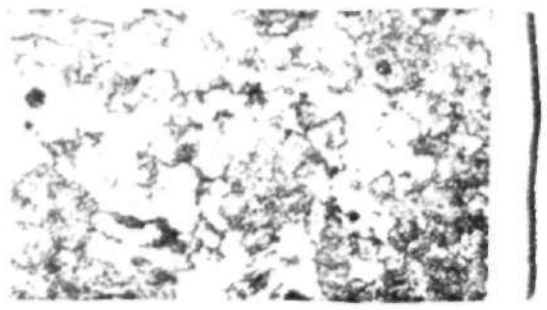


PLATE 5
S/BUTTER-OIL QUENCH

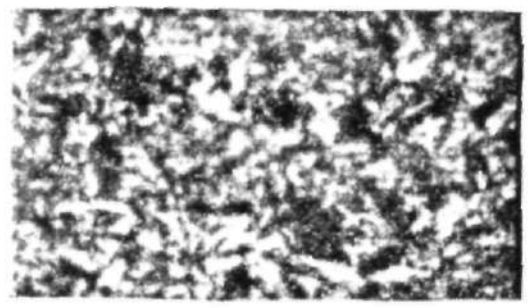


PLATE 6
COLD WATER QUENCH

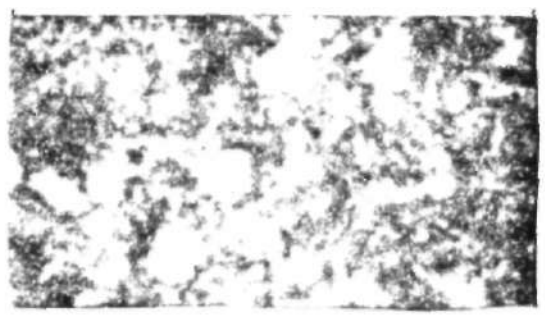


PLATE 7
HOT WATER QUENCH

MICROGRAPH OF 27.90mm DIA. STEEL (0.35% C) ROUNDS QUENCHED IN VARIOUS MEDIA X500, ETCHED IN 3% NITAL.

The structure of all the specimens consists of ferrite (white) and pearlite (dark grey areas). The dispersion is essentially random. The grain size of both ferrite and pearlite are however much finer in the groundnut oil and hot water quenched specimens. However, pearlite predominates in the groundnut oil quenched specimen.

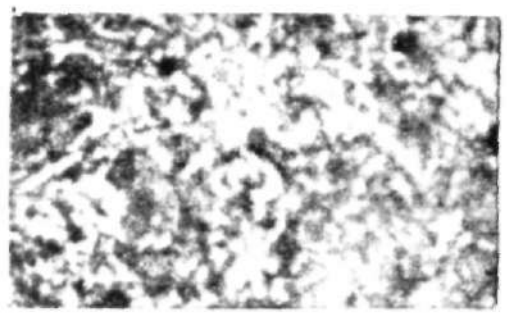


PLATE 8
P-OIL QUENCH

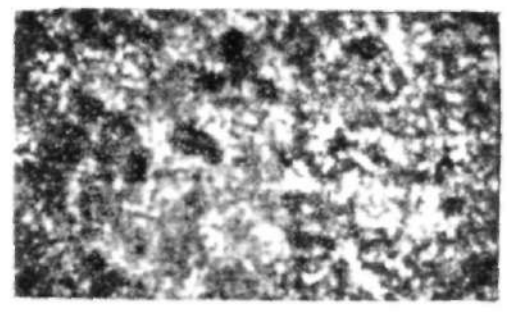


PLATE 9
G/NUT-OIL QUENCH

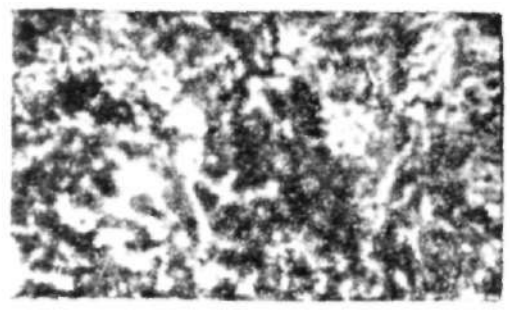


PLATE 10
S/BUTTER-OIL QUENCH

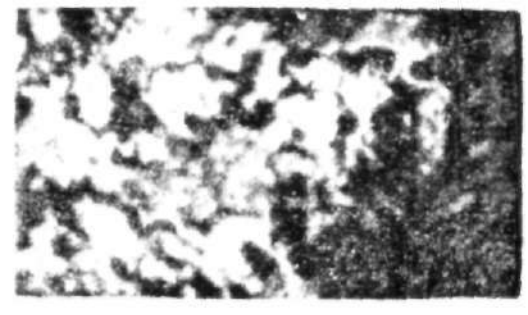


PLATE 11
COLD WATER QUENCH

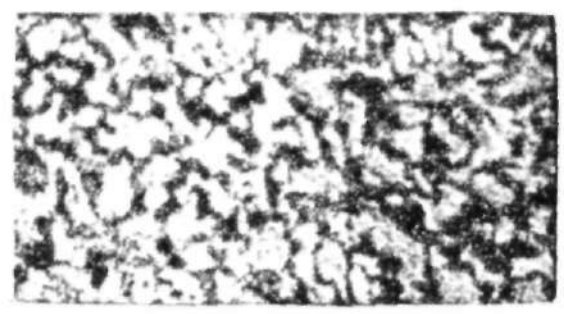


PLATE 12
HOT WATER QUENCH

MICROGRAPH OF 25.40mm DIA. STEEL (0.35% C) ROUNDS QUENCHED IN VARIOUS MEDIA X500, ETCHED IN 3% NITAL

Main structures are essentially ferrite (white) and pearlite (black). The hot water quenched specimen shows pearlite dispersion in a ferrite matrix, the proportion of ferrite being higher. The groundout oil quenched specimen, however has a higher proportion of pearlite and also contains particles of oxide (black dots). Pearlite of varying grain sizes was observed in the cold water quenched specimen micrograph.

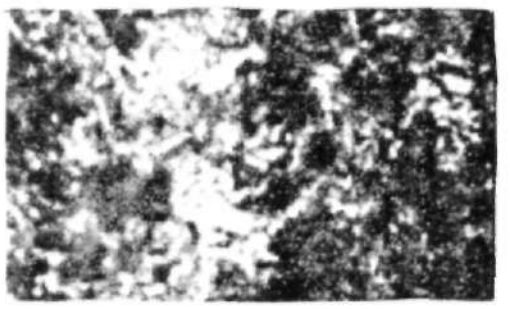


PLATE 13
P-OIL QUENCH

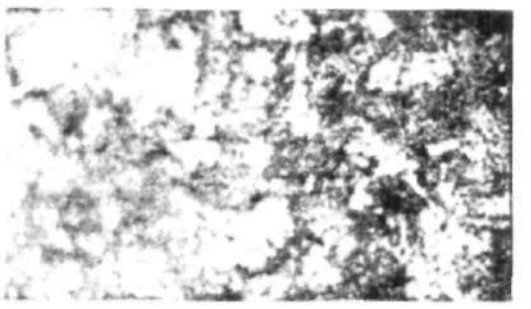


PLATE 14
G/NUT-OIL QUENCH

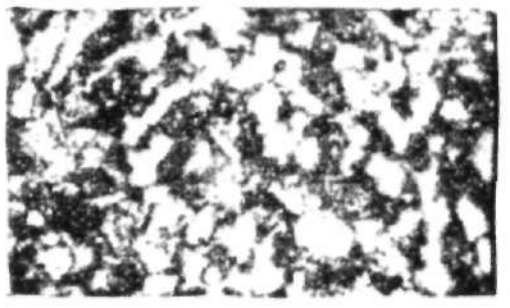


PLATE 15
S/BUTTER-OIL QUENCH

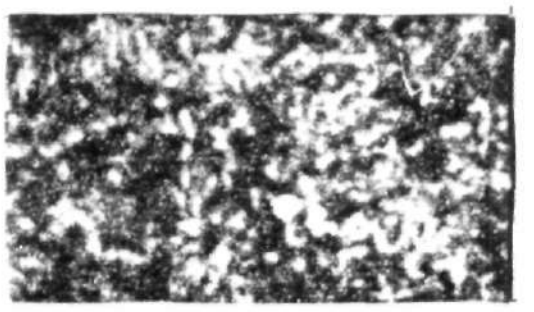


PLATE 16
COLD WATER QUENCH

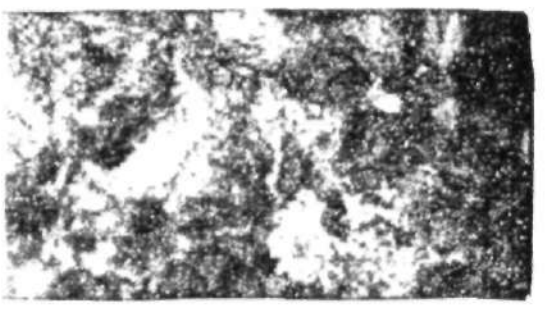


PLATE 17
HOT WATER QUENCH

MICROGRAPHS OF 19.05mm DIA. STEEL (0.35% C) ROUNDS QUENCHED IN VARIOUS MEDIA X500, ETCHED IN 3% NITAL.

Plate 16 (micrograph of the water quenched specimen) shows a dead ferrite (white) - pearlite (black) dispersion and finer grain size when compared with the micrographs of specimens quenched in palm oil, groundnut oil, and shea butter oil (plates 13, 14, and 15 respectively). In all the micrographs, microstructure comprised of ferrite (white) and pearlite (black).

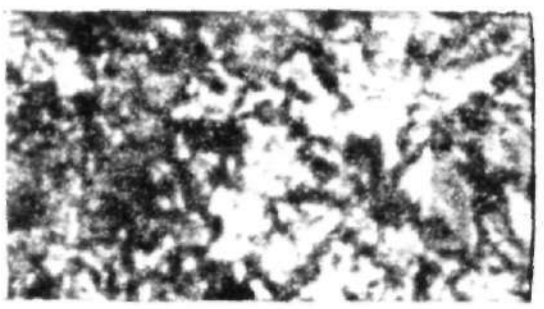


PLATE 18
P-OIL QUENCH

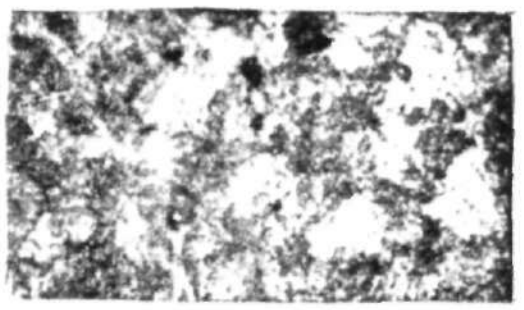


PLATE 19
G/NUT-OIL QUENCH

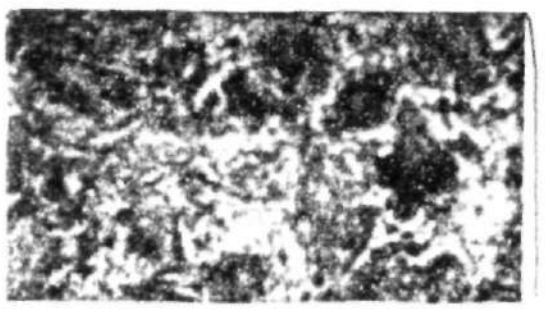


PLATE 20
S/BUTTER-OIL QUENCH



PLATE 21
COLD WATER QUENCH

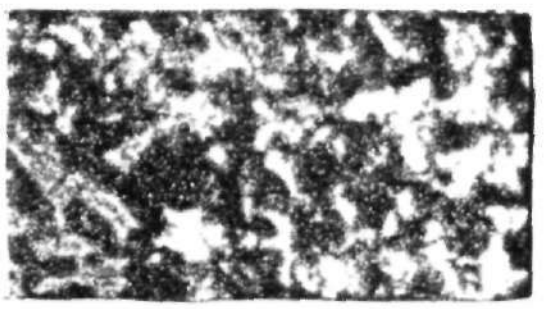


PLATE 22
HOT WATER QUENCH

MICROGRAPHS OF 12.70mm DIA. STEEL (0.35% C) ROUNDS QUENCHED IN VARIOUS MEDIA X500, ETCHED IN 3% NITAL.

Microstructure of all the specimens are essentially ferrite (white) and pearlite (black). Plate 19 shows some particles of oxide (black dots) the shea butter oil quenched specimens show a light area (to the left) which most likely represents a region decarburization as a result of abrasive cutting; it also contains some oxide particles. Plates 20 and 21 indicate onset of elongation of the black constituents, probably upper bainite. The white area is ferrite.

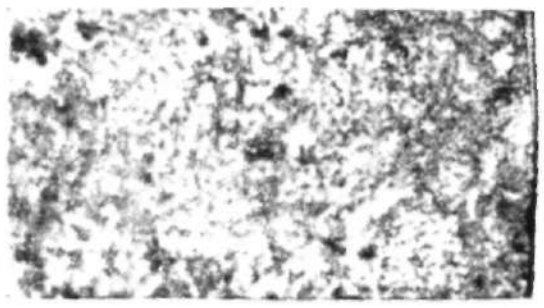


PLATE 23
P-OIL QUENCH

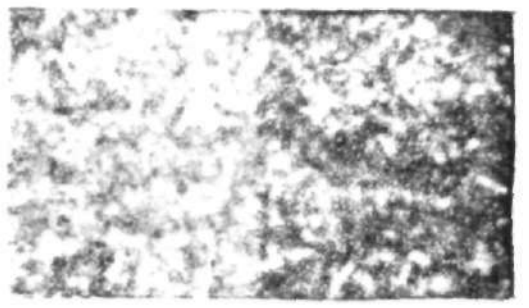


PLATE 24
G/NUT-OIL QUENCH

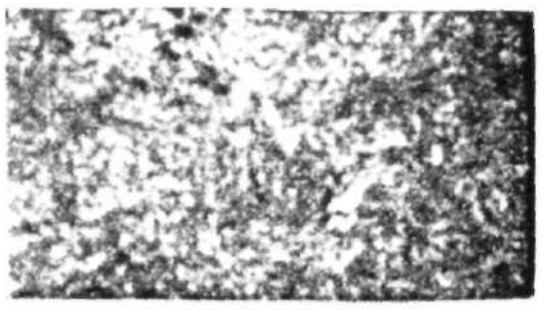


PLATE 25
S/BUTTER QUENCH

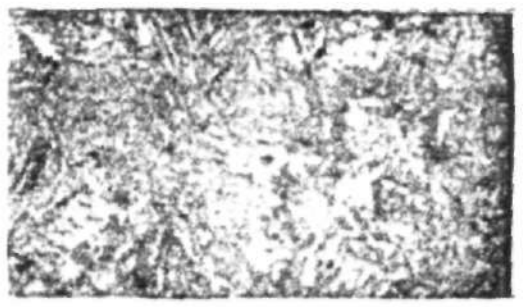


PLATE 26
COLD WATER QUENCH

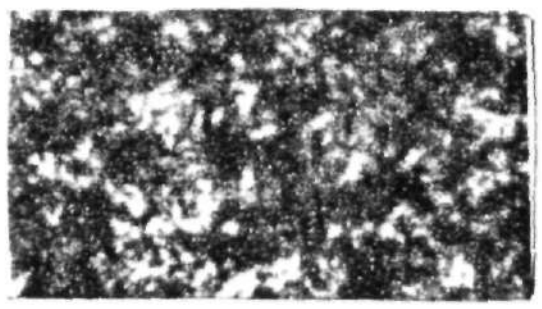


PLATE 27
HOT WATER QUENCH

MICROGRAPHS OF 6.35mm DIA. STEEL (0.35% C) ROUNDS QUENCHED IN VARIOUS MEDIA X500, ETCHED IN NITAL.

Structure in plate 27, the hot water quenched specimen, is mostly unresolved pearlite (black) and some ferrite (white). Micrographs of palm oil, groundnut oil, shea butter oil and cold water quenched specimens reveal some percentage of bainite structures. Traces of ferrite (white patches) can be found in the micrographs of the rest but not in that of cold water quenched specimen micrograph.

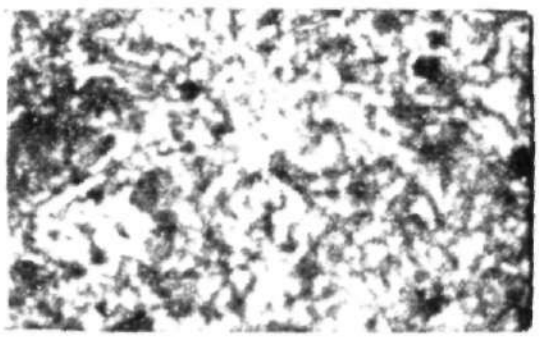


PLATE 28A
BATH TEMPERATURE 28 ^{o}C



PLATE 28B
BATH TEMPERATURE 75 ^{o}C

MICROGRAPHS OF 25.4mm DIA. STEEL (0.355% C) ROUNDS QUENCHED IN PALM-OIL OF DIFF BATH TEMPERATURE, X500, ETCHED IN 3% NITAL

Quenching at a higher bath temperature (75 ^{o}C as against 28 ^{o}C) results in a microstructure with a higher proportion of pearlite (dark region). Finer grain size of the pearlite, compared to the ferrite (white) is also evident in plate 28B

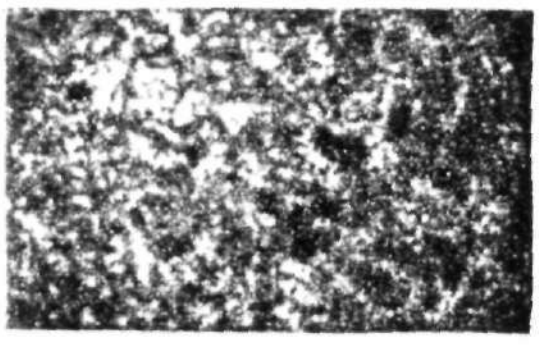


PLATE 29A
BATH TEMPERATURE 26 ^{o}C

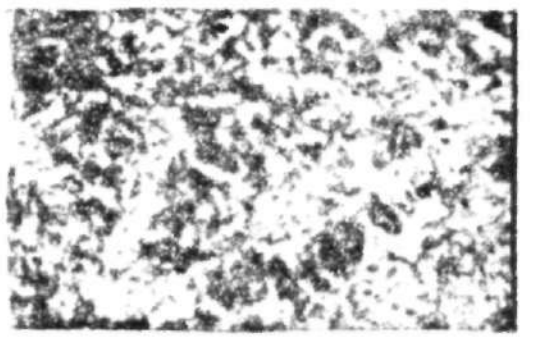


PLATE 29B
BATH TEMPERATURE 65 ^{o}C

MICROGRAPHS OF 25.4mm DIA. STEEL (0.35% C) ROUNDS QUENCHED IN GROUNDNUT OIL OF DIFF BATH TEMPERATURES, X500, ETCHED IN 3% NITAL

An increase in the amount of ferrite formed is observed in the high (65 ^{o}C) bath temperature quenched specimen (plate 29B). However, the pearlite (dark) grain size that were formed are much finer and better dispersed than in specimen quenched in the bath at lower temperatures (26 ^{o}C).

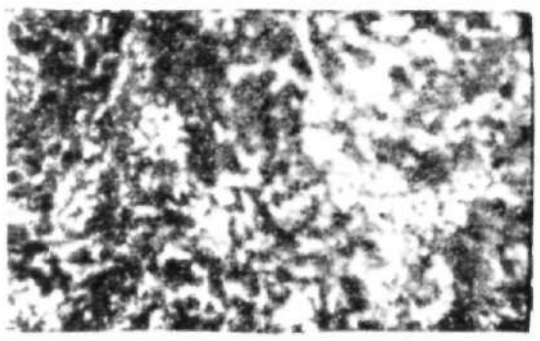


PLATE 30A
BATH TEMPERATURE 29 ^{o}C

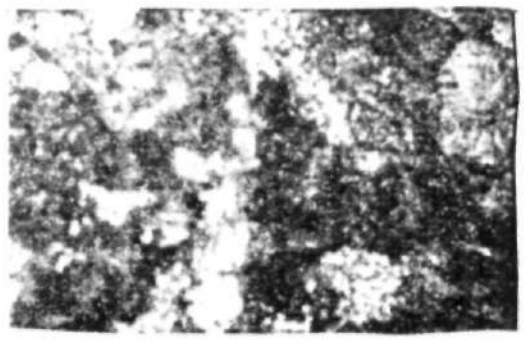


PLATE 30B
BATH TEMPERATURE 80 ^{o}C

MICROGRAPHS OF 25.4mm DIA. STEEL (0.35% C) ROUNDS QUENCHED IN SHEA BUTTER OIL OF DIFF. BATH TEMPERATURES X500, ETCHED IN 3% NITAL.

The hot bath (80 °C) quenched specimen micrograph reveals a structure which comprises of very fine dispersion (dots) in a ferrite matrix (white) and a grey region with acicular structure which is probably upper banite. The matrix is pearlite. The structure in plate 30A, on the other hand is mainly ferrite (white) and pearlite (black).

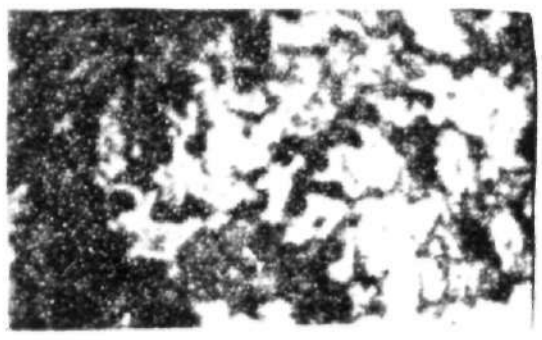


PLATE 31A
BATH TEMPERATURE 26 ^{o}C



PLATE 31B
BATH TEMPERATURE 80 ^{o}C

MICROGRAPH OF 25.4mm DIA. STEEL (0.35% C) ROUNDS QUENCHED IN WATER OF DIFF. BATH TEMPERATURES. X500, ETCHED IN 3% NITAL.

The hot (80 °C) water quenched specimen shows a very high amount of ferrite (white) with very little pearlite (dark). When compared to that of cold water quenched specimen, the proportion of pearlite in the cold water quenched specimen (plate 31A) is also much higher.

Table 4.3 Hardness Value Measurements From Jominy End Quench Test

POSITION FROM END OF BAR AT 1.6mm INTERVAL	VICKERS HARDNESS VALUE
0	440
1	389
2	342
3	304
4	286
5	249
6	220
7	202
8	194
9	195
10	201
11	193
12	188
13	187
14	172
15	175
16	181
17	178
18	180
19	179
20	171
21	168
22	170
23	167
24	165
25	160
26	162
27	163
28	164
29	159
30	156

Table 4.4 Quench Severity Test Results

MEDIA	HARDNESS VALUES AT CENTER OF	
	25.4mm DIA. POSITION	12.7mm DIA PORTION
Palm Oil	179	230
G/Nut oil	171	246
S/Butter Oil	211	226
C/Water	277	359
H/Water	159	212

4.4 Viscosity Test

Viscosity value were determined for all the oils investigated.

Measurement were made at two different temperatures; room temperature (26 ^{O}C) and 60 ^{O}C, in Redwood seconds and given in table 4.5

Table 4.5 Viscosity Values of Various Oils Under Investigation at Room Temperature, 27 ^{O}C and 60 ^{O}C

Oil	Viscosity (Redwood seconds)	
	27 ^{O}C	60 ^{O}C
Palm Oil	245	115
Groundnut oil	225	108
Shea Butter Oil	257	126

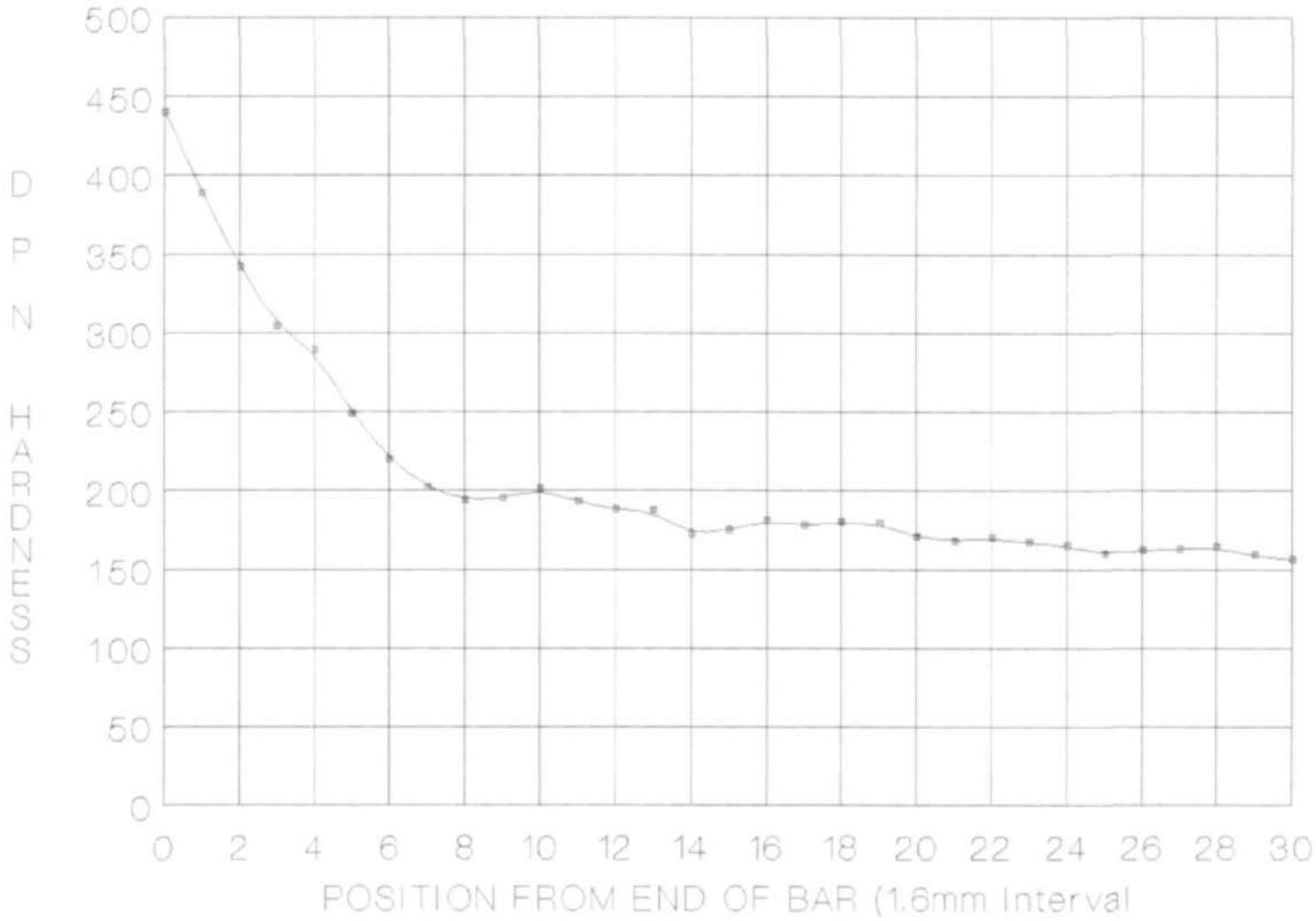


Fig 4.15 Jominy End Quench Test Result

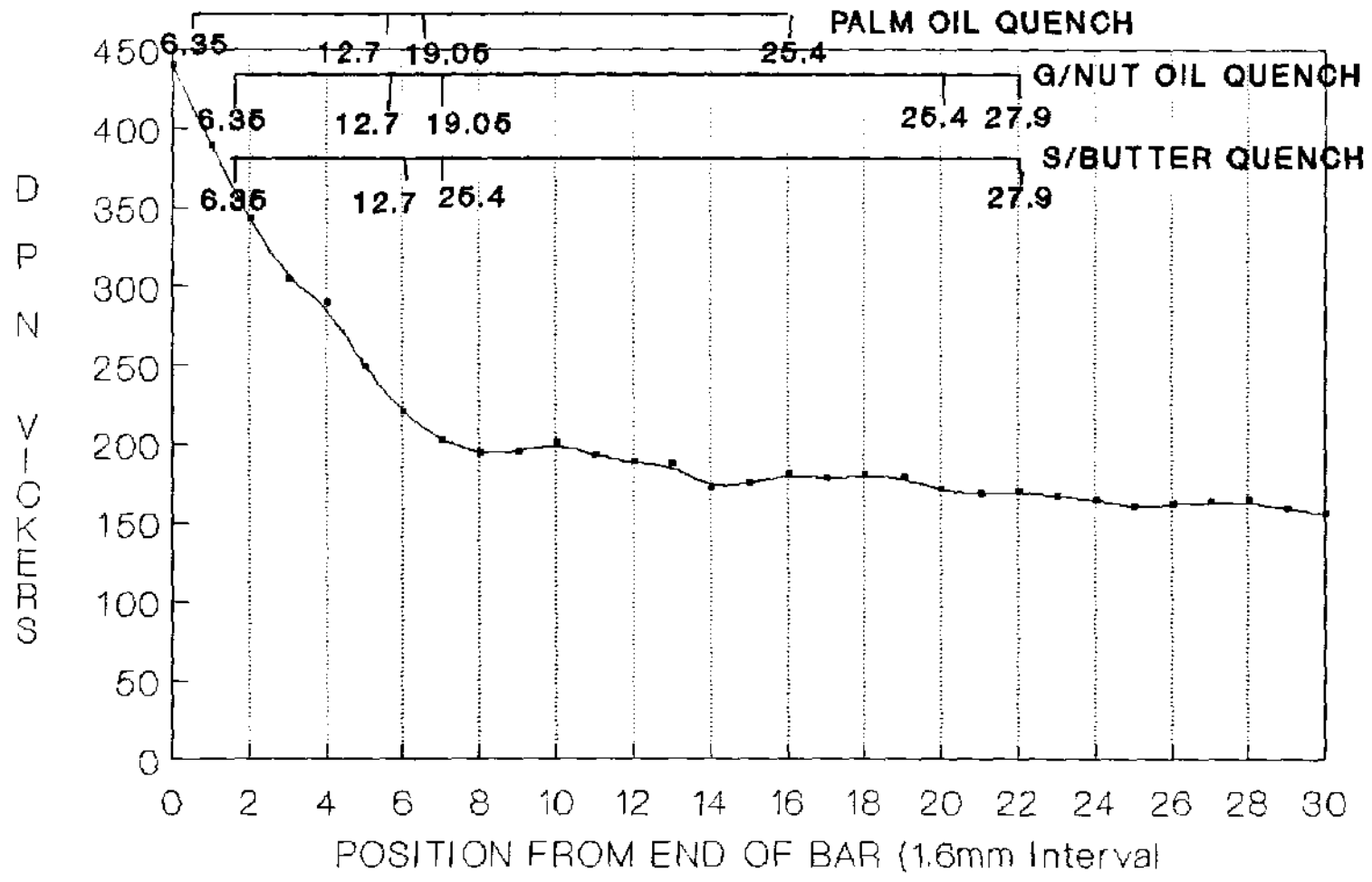


Fig 4.16 Centre Hardness of Oil Quenched Bars and Corresponding Jominy End Quench Distance

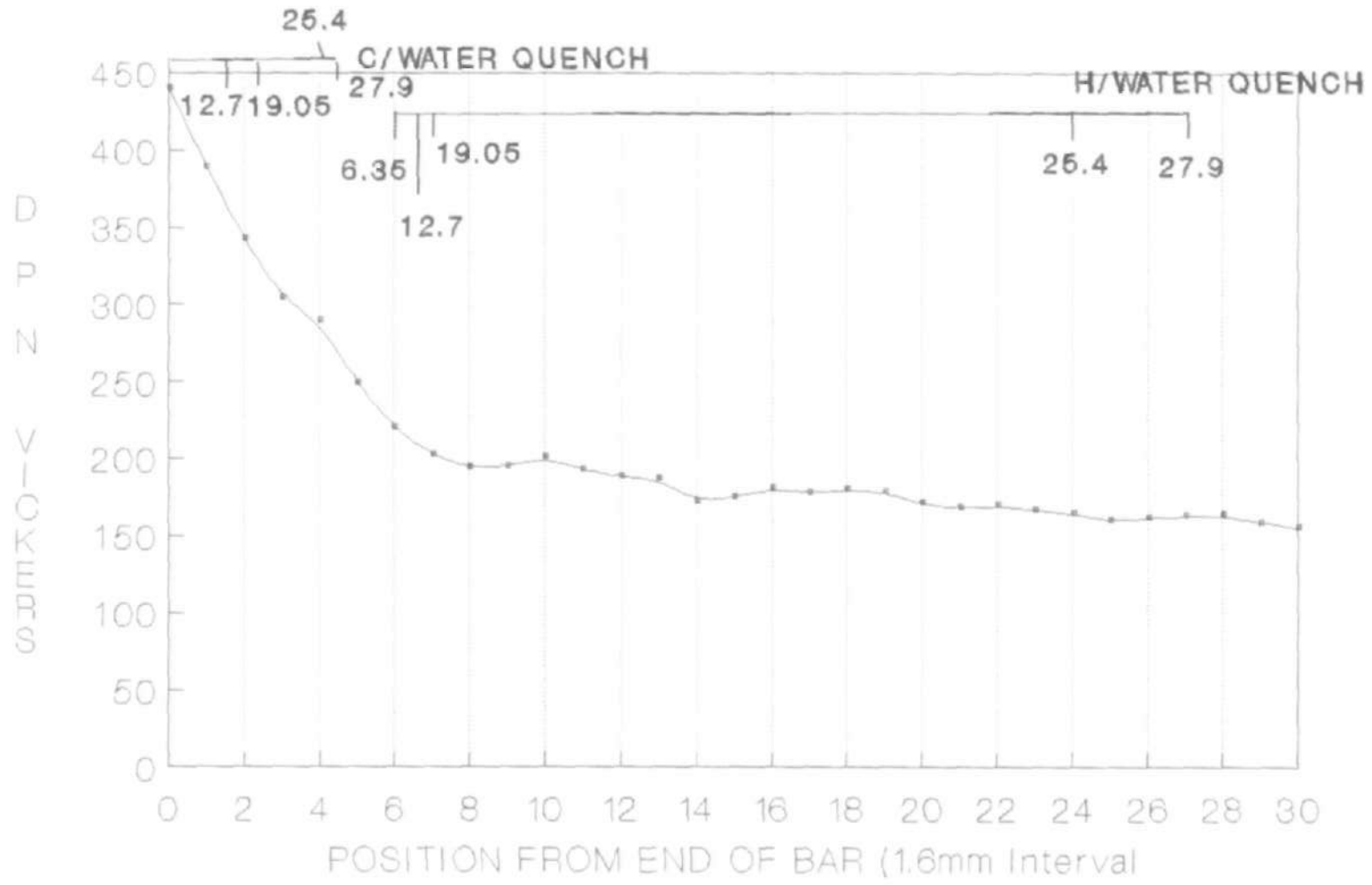


Fig 4.17 Centre Hardness of Water Quenched Bars and Corresponding Jominy End Quench Distances

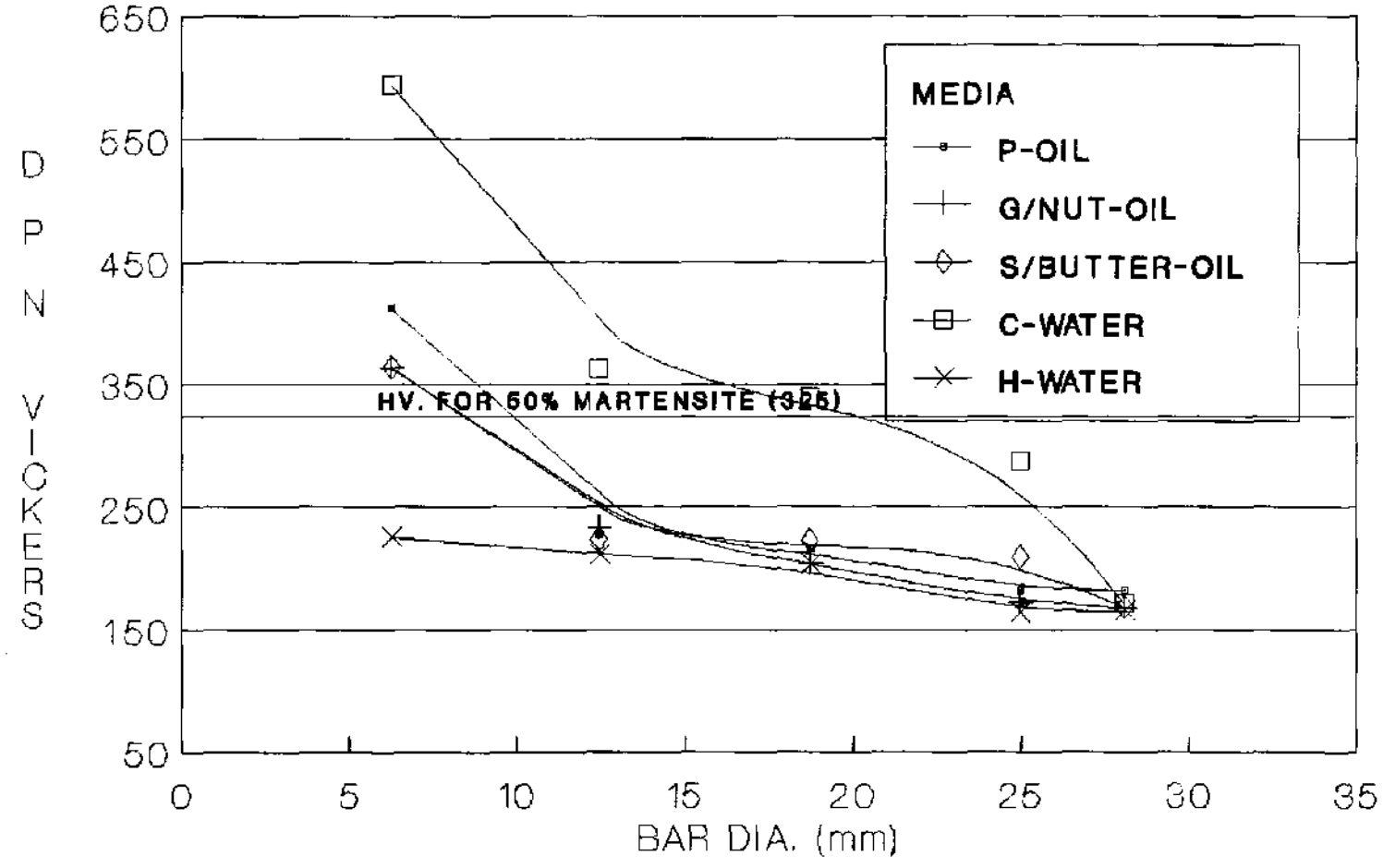


Fig 4.18 Hardness at the Centre of Quenched Steel Bars of Progresively Larger Diameters

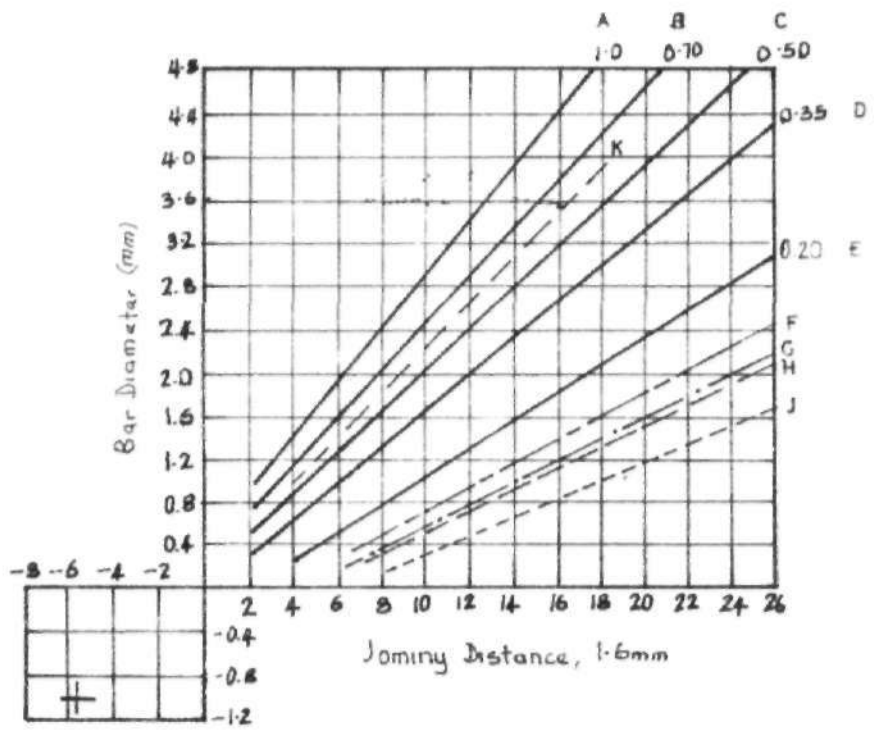


Fig 19 Quench Severity Chart

LEGEND

A: Quench severity line for poor water quench; no agitation.
B: Quench severity line for strong oil quench; violent agitation.
C: Quench severity line for very good oil quench; good agitation.
D: Quench severity line for good oil quench quench; moderate agitation.
E: Quench severity line for poor oil quench; no agitation.

F: Quench severity line for palm-oil quench; no agitation.

G: Quench severity line for shea butter-oil quench; no agitation.
H: Quench severity line for ground nut-oil quench; no agitation.
J: Quench severity line for hot water quench; no agitation.

K: Quench severity line for cold water quench; no agitation.

CHAPTER V

5.0 DISCUSSION OF RESULTS

5.1 Range of conditions

The suitability of fatty base local oils with respect to inducing hardenability potentials in steels during quenching operations was investigated in this research work.

The Grossmann cylindrical test procedure which employs metallurgical response in evaluating quenchants was adopted. Thus, hardness distribution across the traverse section at the mid position of the specimen was used as the major criteria for assessing each oil under investigation. In addition, microstructural examinations of quenched specimens were used to corroborate hardness measurement results.

Review of previous studies on quenching operations revealed the influence of bath temperature, immersion depth, steel composition and several other factors. Accordingly, the variables examined in the present investigation were:

- Quenchant
- Quenchant temperature
- Specimen size.

In a parallel investigation, reappraisal of a locally produced structural carbon steel was carried out by quenching sample specimens from this steel in the investigated oils and in water at various temperatures. This was done to authenticate its hardenability potentials after quenching in water and in these oils and compared with quenching in a standard industrial paraffinic oils.

The hardening characteristics of the steel under investigation was determined from the end quench test in line with Jominy end quench test procedure. Results obtained were then correlated with those obtained from the Grossmann test to estimate the size of bar which will fully or partially respond to heat treatment when quenched in any particular oil.

5.2 Assessment Of Results

5.2.1. Hardness Measurement

The 50% martensite criterion as established by Grossmann et al was employed in this research as a measure of hardened zone. For this percentage of martensite, the corresponding hardness value for a 0.35% carbon steel is 325 Vickers (Diamond) pyramid hardness number (DPN) as established by several researchers.

5.2.1.1 Water Quenching

From the hardness measurements, both the 6.35mm and 12.7mm diameter specimens hardened through when quenched in cold water (26 ^{O}C) but not so in hot water. The centre hardness values obtained in the cold water quench was 597.7 DPN for the 6.35mm diameter steel at about 0.795mm from the specimen surface while the 12.7mm diameter specimen gave 377 DPN.

Hardness traverse for hot water - quenched specimens indicates a decrease in surface-to-centre hardness. This shows a slow cooling rate by hot water. In addition, hardness values obtained in hot water quenched specimens portray, essentially, a ferrite-pearlite microstructure on

the surface of the steel as revealed in all the micrographs (plates 7, 12, 17, 22, 27). Consequently, cold water quench gives a much more severe quench than hot water.

Results obtained is in agreement with established results for this grade of steel thus establishing the quality of the steel with respect to its response to hardening. This also proves adherence to good quenching procedure and reliability of results obtained. The results thus serve as basis for comparative analysis with results obtained from oil quenching since the same operating condition prevailed.

5.2.1.2 Fatty oil quenching

Only the 6.35mm diameter specimen attained the desired 50% martensite hardness when quenched in all the investigated fatty oils. The maximum hardness recorded was on the specimen quenched in palm oil, with a value of 465 DPN at the surface.

The hardness traverse across the diameter of these specimens were essentially gradual for all the quench media (figure 4.10). This indicates a protracted film boiling stage which reduces fast heat extraction and consequently substantial transformation of the austenite. Thus, the commencement of the nucleate boiling does not generate much martensization hence the gradual increment in the centre-to-surface hardness.

Considering the 6.35mm diameter specimen, palm oil proved to be the most effective quenchant. The centre hardness recorded on the palm oil quenched specimen was not only higher than those obtained at the centre of other

specimens quenched in the remaining oils, its surface hardness value was also greater. The centre and surface hardness values were 412 and 465 respectively for palm oil quench, 364 and 411.5 for shea butter oil quench, and 364 and 409 for groundnut oil quench.

Shea butter oil was most sensitive to the effect of mass of specimen on the depth of hardening. It showed less abrupt centre-to-surface hardness variation irrespective of the geometry of the quenched specimen (figure 3)

From the hardness traverse plots for these oils, it can be seen that groundnut oil and shea butter oil gave similar results for which the 6.35mm diameter bar and the 27.9mm diameter bar had respective centre hardness values of 1.5 (2.4mm) and 22 (35.2mm) of the end quench distances. However, it was observed in figure 4.16, that the equivalent jominy distance for the 25.4mm diameter specimen differed a great deal with shea butter oil having 7 and groundnut oil having 20.

The equivalent jominy distances for hot water quenched bars are mostly in the unhardened region; conversely, those for cold water quenched bars are within the hardened region except in the case of the 27.9mm diameter bar which had an equivalent end quench distance of 4.5 (7.2mm).

5.2.2 Metallographic Observation

The microstructure observed in quenched specimens verifies the hardness measurement results. The inability of most of the specimens to attain the desired critical hardness

(325 DPN) is reflected in the "after quench" microstructure. Essentially, all the microstructures observed were ferrite and pearlite.

The variation in hardness observed in specimens of the same size quenched in different media stems from the relative proportion of ferrite-to-pearlite, and also their dispersion. In addition, variation of the grain size of the microconstituents also contributes to the difference in hardness since the properties of pearlite depends largely on the carbide spacing.

Banite was observed in the structures of the 6.35mm diameter specimens quenched in all the oils and cold water. In the case of hot water, unresolved pearlite and some ferrite grains were the only microconstituents, indicating the inadequacy of hot water as a quenching medium, at least, for plain carbon steels.

Based on the microstructural observation and hardness measurement, it can be deduced that palm oil proved most effective followed by shea butter oil and then groundnut oil, for 6.35mm diameter specimens quenched in different oil media investigated.

5.3 Bath Temperature Effect

In the range of bath temperature considered (26 ^{O}C - 80 ^{O}C), all the results show an increase in hardness measurement with higher bath temperature. Palm oil and groundnut oil - quenched specimens show gradual increase in centre-to-surface hardness while shea butter oil quenched specimen gave a much

more steeper hardness gradient after quenching in a higher temperature bath. In water quenching however, lower hardness measurement was recorded at 80 ^{o}C, while in cold water quenching, higher hardness value was obtained (figure 4.11, 4.12, 4.13, 4.14).

Hardness measurement from the water quenched specimens and the observed microstructure conform with the expectation. In cold water quenching, the film boiling stage is unstable, and as such, nucleate boiling manifests early thus improving heat extraction which results in better hardenability. The stability of the film during boiling improves as temperature of the bath increases thus impairing the cooling power of the water.

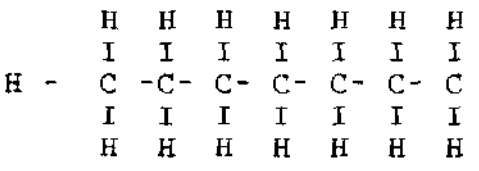
The degree of undercooling (100-T_s) which determines the stability of the film boiling stage acts as the singularly most important parameter which influences the difference in quenching results obtained in cold water and hot water. As a result of the stability of film boiling in the hot (80 ^{o}C) water quenching operation, the transition temperature from film boiling to nucleate boiling became depressed thereby reducing the ability of the steel to form martensite.

The centre hardness values obtained on the higher bath temperature quenched specimens were 195 DPN for palm oil, 176 DPN for groundnut oil and 256 DPN for shea butter oil. With similar specimen geometry and quenching at low bath temperature of 23 oC, the DPN were 182, 172, and 209 respectively for palm oil, groundnut oil and shea butter oil.

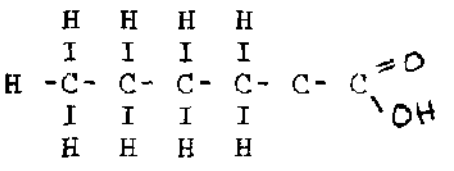
Examination of the micrographs showed better dispersion of finer pearlite grains in the higher-bath-temperature quenched specimens except in the case of groundnut oil. This explains the observed increase in hardness in the palm oil and shea butter oil - quenched specimens.

With rated industrial quenchants, the duration of film boiling varies very little with temperature and as such not much variation in the hardness traverse is expected with an increase in bath temperature. This, however, is not the case with the fatty oils investigated. An increase in bath temperature improves cooling power of the oil.

The reasons for the disparity can be derived from the nature of the oils. Although the molecule of fatty acids are similar to the paraffins (a typical industrial quenching oil), they have, in addition, a polar (active) group which is made up of an OH (hydroxyl) and an O (oxide) to form the unsaturated carboxyl group. The structures are as shown below:



Paraffin Oil
(Saturated Molecule)



Fatty Oil
(Unsaturated Molecule)

An increase in the oil temperature results in pyrolysis of the oil to form stable chemical species. This ageing gives rise to a reduction in the cohesive adhesion between the molecules. The result is that the duration of film boiling

is reduced because the extensive film of fatty acid molecules on the metal surface no longer prevails.

It is also possible that deposition of products of pyrolysis on the metal surface would also generate extra nucleation sites for bubble formation thus enhancing the nucleate boiling stage and consequently higher rate of cooling.

5.4 Effect Of Viscosity

The average viscosity of each of the oil under investigation is as given in Table 4.5. Shea butter oil was found to have the highest viscosity value of, 257 (Redwood seconds) followed by palm oil with a value of 245 and then groundnut oil, having 225 all at 27 ^{o}C. The same trend was observed when the viscosity was measured at 60 ^{o}C. Variation of viscosity with temperature justifies the temperature dependence of the characteristics of this oils as reflected in the results obtained in different bath temperatures.

As expected, the higher the viscosity, the lower the cooling rate, and consequently the smaller the probability of martensite formation in the quenched steel. This is due to the considerable thermal resistance that exists in viscous oils. Consequently, the transition temperature between film and nucleate boiling on one hand and between nucleate boiling and convection on the other hand will be determined or influenced by the viscosity of the oil.

The order of severity of quench of the oils investigated vis-a-vis the viscosity does not completely agree. On the basis of the quench power of the oils investigated, they can be ranked in decreasing order as palm oil, shea butter and groundnut oil. However, viscosity values in this investigation give a rating, in decreasing order, as shea butter, palm oil and groundnut oil. The most plausible explanation is that, while viscosity affects the cooling power of the oils, the degree of unsaturation of the fatty oil has an over bearing effect. The degree of unsaturation is indicative of the amount of reactive radicals present and is measured by the content of iodine in the oil.

The proportion of the reactive radicals determines the degree of molecular adherence of the oil to the metal surface in the form of fatty oil film. Thus the higher the reactive radical content, the stronger the film. The heat extraction from the metal surface is consequently impaired.

The iodine content which is the reactive radical present in the oils investigated, is in agreement with this observation. Palm oil, with the least iodine value showed the highest cooling power while groundnut oil with the highest iodine value gave the least severe quench.

5.5 Effects of Oil Composition on Quenching

The structure of fatty oils bears structural resemblance to that of paraffinic mineral oils, which are known generally as quenching oils. Fatty oils, unlike paraffins, are unsaturated. They contain unsaturated fatty acids in

addition to (Polar) carboxylic group, which are also reactive. The unsaturated content of the fatty oils (acid and caboxylic group) usually contributes to a strong molecular adhesion to a metal surface which results when the metal is brought into contact with the oil.

5.5.1 Fatty Acid Composition

Fatty oils contain saturated and unsaturated acids. The saturated ones are usually myristic, palmitic, stearic, caprytic, capric, lauric, arachidic, behavic and ligonoceric. The unsaturated acids are mostly oleic and linoceric acids. The saturated acids in the presence of the caboxylic group are usually responsible for the molecular adhesion to the metal surface which is then complimented by the unsaturated fatty acids to make the adhesion to the metal surface stable.

5.5.2 Iodine Value

Iodine value is a measure of the degree of unsaturated fatty acids in the oil. It indicates the amount of reactive radicals present in them and by extension, the stability of the fatty acid molecule film that forms on a metal surface. Groundnut oil has the highest iodine value (106.01), while palm oil has the lowest (64.50). The relative content agrees well with the observed heat extraction properties of the oils. Groundnut oil with the highest iodine value rated least effective as a quenchant due to the stability of the film. On the other hand, palm oil gave the best quench as it had the least stable film due to its low iodine value.

5.5.3 Phosphorus Content

Phosphorus reacts with metal surfaces to form metallic soaps. The phosphorus content in the oils under study, though small, must have served to buffer the flow of heat from the metal surface to the fluid. The trend in percentage content variation of the element is reflected in the order of the cooling power of the oils investigated.

5.5.4 Free Acid Content

Free acid content of any fatty oil is a measure of the stability of the oil. It indicates the ease with which the oil will deteriorate (i.e. turn rancid) on exposure to the atmosphere. Of all the oils investigated, shea butter oil is likely to deteriorate the fastest based on the percentage free acid content in the oil (14.71 - 52.36). The most stable oil was found to be groundnut oil with a 0.11 - 0.64 percent free acid content.

5.6 Oils Rating Based On Hardness Traverse

Hardness values across the traverse section show that palm oil rates highest with respect to cooling power. Shea butter oil and groundnut oil are comparable, though shea butter oil is a little better than groundnut oil. This observation is in agreement with the obtained microstructures as revealed in Plates 23, 24 and 25. The hardness induced factors such as the refined ferrite-pearlite structure, are found to be most prominent in the palm oil quenched specimen (plate 23). This is closely followed by shea butter and groundnut oil respectively.

Adopting the 325 DPN for 50% martensite composition, it was found (figure 4.18) that the diameter of bar that will just harden to 50% martensite at the centre when quenched in these oils are approximately 10.2mm for palm oil quench, 9mm for groundnut and shea butter oil quench, and 21mm for cold water.

Quench severity evaluation of individual medium gives the oils as having H values of 0.12 - 0.15 palm oil and 0.08 - 0.18 shea butter and groundnut oil

The quench severity evaluated for cold water is 0.03 - 0.05 hot water is 0.6

Comparing these values with established data, Table 1.2, the results for the oils fall bellow the class of "poor oil quench" without agitation (H-value 0.25 - 0.30), while the hot water - quenched specimen could as well be air cooled (with strong air agitation).

At elevated temperature, the oils collectively gave better results. However, the improvement in performance was highest with shea butter oil. Palm oil showed a 6.7% increase in centre hardness value while shea butter and groundnut oil gave 18.4% and 2.3% respectively

CHAPTER VI

CONCLUSION

A quantitative description of the factors responsible for the cooling properties of the investigated fatty oils, namely, cooling power in respect of the hardenability of plain carbon steel has been given. The cooling power has been related to size of the piece and quenching medium.

Based on ASTM designation, the range of composition (0.3%C - 0.6%C) in which the hot rolled steels lie is defined on the low side by minimum strength requirement and on the upper side by hardenability.

For many important applications, however, subsequent processing, such as quenching are often applied to achieve better strength and improved toughness. Considerable attention has therefore been given to raise strength without increasing the alloy content.

Previous work in this aspect has shown that quenchants such as water, brine and caustic soda, which can be used to stimulate substantial hardening in carbon steels might also distort or induce cracks in the metal. Quenching mineral oils on the other hand, are inadequate with respect to drasticity.

Fatty oils, were investigated in this research work, as alternative quenchants to the aforementioned.

The main difference between fatty oils and mineral oils lies in composition; while fatty oils are polar substances with long carbon chains containing one or more chemically active group, mineral oils are stable with a saturated

molecule. In general, one can state that the difference in the quenching behaviour of the oils lies in the extent of saturation of the oils which is measured by the iodine content of the oil.

The fatty oils compare favourably with mineral oils and substantial martensitic hardness can be obtained if the size of steel being quenched in the fatty oil is small.

In contrast to mineral oil, bath temperature has substantial influence on the quench properties of fatty oils.

Quench severity values of the oils investigated can be used to predict the rate of energy transfer in objects of various sizes and composition. Such predictions can be derived by estimating the Biot (Bi) modulus.

Essentially, structures obtained in the size and material used were ferrite and pearlite, except in the case of the 6.35mm diameter in which banite was observed. It was however observed that grain size and proportion of ferrite were smaller as the sizes progressively grew smaller. The improvement of hardenability was thus derived mainly from the finer and therefore stronger ferritic - pearlitic structure rather than martensite. The grain refinement resulting from quenching of carbon steel in fatty oil is thus worthy of further investigation.

Results obtained from the test of the hardenability properties of carbon steel produced in Nigeria have shown favourable response to quenching in locally produced fatty base oils.

Further study of the quench properties of these oils is however, necessary for fuller understanding of the effect of agitation on quench severity.

In addition it is recomended that the mechanical properties of the steel after quenching should be studied to determine how they are affected by the quenching operation and areas in which the steels, with enhansed properties, could be addvantageously put to use.

Finally alloy steels should be subjected to treatment in these oils so as to realize their full potentials and also determine their suitability.

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