WHAT IS SINTER-HARDENING?

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Abstract

The mechanical properties of ferrous powder metallurgy (P/M) materials are directly related to their density and microstructure. Many *PIM* parts are heat treated, in a secondary operation, to develop a tempered martensitic microstructure either in a surface layer, or throughout the part. The need for a secondary quenching operation may be avoided by "sinter-hardening" the parts.

Ferrous P/M materials with sufficient hardenability will develop microstructures containing significant percentages of martensite in the as-sintered condition. Accelerated cooling · techniques for sintering furnaces have been developed which permit larger parts to be sinter-hardened, or materials with lower hardenability to be Used to produce sinter-hardened parts with smaller cross-sections.

The difference between hardness and hardenability will be explained and a review presented of how the alloying method selected for ferrous P/M materials influences hardenability. Examples of sinter-hardenable materials will be provided and the benefits and disadvantages of the sinter-hardening process will be discussed:

Introduction

In the as-sintered condition, the microstructure of most hypoeutectoid ferrous powder metallurgy (P/M) materials consists of ferrite and pearlite - Figure 1. The relative amounts of ferrite and pearlite in the microstructure depend on the combined carbon content of the material - Figure 2. The spacing of the pearlite lamellae depends on the rate at which the material has been cooled; for faster cooling rates the lameliar structure of the pearlite becomes more difficult to resolve. Using the "Lever Rule" an estimate of the combined carbon content of plain carbon steels may be obtained from the relative amounts of ferrite and pearlite in the microstructure -Figure 3.

P/M carbon steels may be quench-hardened and tempered. This involves heating the component to a temperature in the austenitic region of the phase diagram (Figure 4) 1 and quenching it in an agitated oil bath followed by a stress relieving or tempering treatment. In the heat treated condition the materials have increased strength and hardness but possess less ductility. After quench-hardening and tempering, the microstructure of the material no longer consists of ferrite and pearlite. It is transformed into a structure referred to as martensite -Figure 5.

There is a limit to the cross-sectional size of a carbon steel part that can be transformed throughout to martensite after quench-hardening. In order to increase the size of parts that can be through hardened, and to increase the strength and toughness of heat treated steels, metallurgists have developed various alloy steels.

A review will be made of the alloying methods used in ferrous P/M and the response to heat treatment of alloys produced by the various methods. The microstructure of some materials with higher alloy content consists predominantly of martensite even in the as-sintered condition. Such materials are referred to as being "sinter-hardenable". While sinter-hardening of material.' such as FLC-4608 has been a commercial practice for many years, it is only recently that the term "sinter-hardening' has been applied to the process. Sintering furnaces equipped with enhanced cooling capability also have been produced for many years. However, there is currently a greater interest in fitting furnaces with such a capability and there are more furnace makers offering such an option.

After a review of the alloying methods used in ferrous powder metallurgy the term hardenability will be introduced and the difference between hardness and hardenability will be explained. The effect of various alloying additions on hardenability will be discussed and the difficulty of producing and using alloys that contain specific alloy additions will be addressed.

The different demands of a material targeted for high hardness will be compared with those of; sinter-hardenable material where the need is for a combination of strength, hardness, and ductility.

Alloying Methods in Ferrous Powder Metallurgy
The mechanical properties of ferrous P/M materials are directly related to their microstructure and the size, distribution, and morphology of the porosity they contain. Alloying additions are made to develop specific material performance characteristics such as hardenability or toughness in the heat treated condition. However, the manner in which the alloys are constituted has a significant effect on the porosity and microstructure of the final sintered product. There are four main alloying methods for ferrous P/M materials and materials may be classified by the manner in which the alloy has been constituted:

- Admixed - The alloying additions are made to the iron powder base in the form of elemental or ferroalloy powders. This is the least expensive and most commonly used alloying method. Since the iron powder base is unalloyed when the mix is pressed, admixed materials retain most of the compressibility of the iron base. The degree of alloying is limited by the mutual diffusivity of the alloying elements and iron at the sintering temperature, and the resulting microstructures are chemically heterogeneous. This type of material is also subject to powder segregation and dusting during handling and pressing.

- Partially Alloyed (Diffusion Alloyed) - The alloying additions are diffusion alloyed to the base iron particles such that the compressibility of the base iron is essentially retained. These materials are often referred to as diffusion alloyed. The powders are highly compressible, and yield heterogeneous microstructures consisting of lightly

alloyed particle cores with a continuous network of more highly alloyed interparticle bonds.

- Prealloyed - The alloying elements, except for carbon, are added to the melt before atomization. This results in homogeneous microstructures and uniform hardness even on a microindentation hardness level. Solution hardening of the powder particles by the alloy additions generally decreases the compressibility of the powders compared with admixed and partially alloyed materials. However, prealloyed powders that use molybdenum as their principal alloy addition have been developed with compressibility's approaching those of iron powders [2].

- Hybrid Alloys - With the advent of highly compressible prealloyed powders, materials have been developed based on additions to these powders [3,4]. For example, materials in widespread use are based on an addition of 2 weight percent (w/o) nickel and 0.4 - 0.6 w/o graphite to a prealloyed powder with 0.85 w/o molybdenum as the principal prealloyed addition. Chrome-manganese P/M steels have been introduced based on additions of high carbon ferrochromium and ferromanganese powders (less than 20 μm particle size) to a prealloyed steel (0.85 w/o molybdenum)[5]

   It is the hardenability of the sintered material as the compact exits the hot zone of the sintering furnace that, for a given cooling rate, controls the microstructure that will be developed. The alloying additions must be in solution in order to contribute to hardenability. Admixed, partially alloyed, and hybrid materials depend on diffusion processes during sintering to effect alloying. Therefore, in general, prealloyed powders have better hardenability than admixed, partially alloyed, or hybrid materials. While some hybrid Cr-Mn steels have hardenabilities as good as   prealloyed materials they need to be high temperature sintered (2300°F {1260°C}).

The Difference Between Hardness and Hardenability

There is a clear distinction between the hardness and the hardenability of a material. Hardness refers to the resistance of a metal to plastic deformation, usually by indentation. Indentation hardness may be measured by various hardness tests (Rockwell, Brinell, Vickers). The hardness measured on a porous P/M material is referred to as an apparent hardness. For a given microstructure, the apparent hardness of a ferrous P/M material is related to the density of the material; apparent hardness increases as density increases.

Hardenability refers to the relative ability of a ferrous alloy to form martensite when cooled from a temperature in the austenitic region of the phase diagram. Hardenability is commonly measured as the distance below a quenched surface where a metal exhibits a specific hardness (50 HRC for example) or a specific percentage of martensite in the microstructure.

The carbon content of a steel affects the hardness of a martensitic microstructure as does the percentage of martensite in the microstructure [6]. Figure 6.

Hardenability Measurement

The Jominy test may be used to measure the hardenability of a material following ASTM method A 255 7. A schematic of the ap6paratus for conducting the Jominy end-quench hardenability test is shown in Figure 7 . In the test, the test specimen, a 1.00 inch (25.4 mm) diameter bar 3 inches to 4 inches (76 mm to102 mm) in length, is water quenched from one end face. The bar from which the test specimen is made must be normalized before the test specimen is machined. The test involves heating the test specimen to the appropriate austenitizing temperature and then transferring it to a quenching fixture designed such that the specimen is held vertically 0.5 inch (12.7 mm) above an opening through which a column of water can be directed against the bottom face of the test specimen - Figure 7. The end nearest the water jet experiences the fastest cooling rate while the end of the specimen remote from the water column cools slowly in air. Various positions along the bar are cooled at rates intermediate between these two extremes. After the specimen has been quenched, parallel flats are ground 0.015 inch (0.38 mm) deep 180° apart on the cylindrical surface of the Jominy bar. For wrought and cast materials, Rockwell C hardness indentations are made at intervals of 1/16 of an inch (1.6mm) along the bar (1/32 inch intervals {0.8 mm} for carbon steels) and the data are reported in the form of a Jominy hardenability curve as illustrated in Figure 8[8].

A recent study showed that the hardenability of ferrous P/M materials, as measured by the Jominy distance to 50 HRC or 30 HRC (apparent hardness converted from 76 HRA or 85 HRA respectively), or the depth to 50% martensite, increases with increasing density 9. The Jominy hardenability curves for an FL-4805 material are shown to be a function of the density of the material - Figure 9. The Jominy data for porous P/M materials reflect two characteristics of P/M materials. The hardness measured is an apparent hardness that is dependent on the density of the material. In addition, the thermal conductivity of the P/M material also is a function of the density of the material - see Figure 10. During Jominy hardenability testing, not all P/M materials have apparent hardness values that can be measured on the Rockwell C scale. That is why hardness measurements for the Jominy testing of P/M materials are made using the Rockwell A scale. The Rockwell A scale is selected because it provides a continuous scale covering the entire range of hardness observed during the Jominy testing. The Rockwell A scale uses a load applied with a 60 kg mass while the Rockwell C scale uses a load applied with a 150 kg mass.

The recent development of an improved technique for making TTT diagrams for P/M alloys will make it easier to develop transformation data for P/M materials ~0. In conjunction with the development of additional Jominy data for P/M materials this should enhance the P/M hardenability database.

Alloying Methods - Effect on Hardenability

While carbon steels may be heat treated to increase their hardness and strength, there is a limit to the size of a carbon steel part that can be transformed to martensite throughout its cross-sectional area. This reflects the limited hardenability of plain carbon steels. In wrought steels, alloy additions are made to improve the hardenability and the subsequent heat treatment response of the material. Alloy additions have a similar effect in P/M materials. However, there are some practical limitations and

consequences of the fact that most ferrous powders are produced by water atomization of molten steel. Elements that have a high oxidation potential are not good additions to ferrous powders because once formed their oxides are not reduced during subsequent annealing of the powder Reduction of oxides formed by elements such as chromium, manganese., silicon, and aluminum requires higher temperatures and lower dew points than those normally used to produce prealloyed Low-alloy powders[11] . Figure 11. The annealed cake produced at such a high temperature is difficult to disintegrate back to a powder without excessive work hardening of the material; the material therefore requires re-annealing to make it sufficiently compressible for die compaction. The re-annealed cake is also hard to disintegrate. It is therefore difficult to produce prealloyed chromium-manganese P/M steels with Low oxygen contents and good compressibility and, in general, the chromium-manganese steels popular in wrought steel metallurgy are not found in ferrous powder metallurgy. The alloying additions most used in P/M are nickel, molybdenum, and copper. The oxides of these elements are reduced during powder annealing and compacts produced from such materials may be sintered at the industry's normal sintering temperature of 2050°F (1120°C) without the problem of re-oxidation.

Alloying additions to steels may be classified into various categories such as ferrite strengtheners, austenite stabilizers, and carbide formers. The influence of alloy additions on the equilibrium phases present in steels is recorded in phase diagrams. These, however, reflect the microstructural phases that will be present in a material under equilibrium conditions. They do not provide an indication that upon rapid cooling a steel will transform to martensite rather than a ferrite/pearlite microstructure. In order to understand the effect of alloy additions on the heat treatment response of steels, metallurgists have developed time-temperature-transformation (TTT) diagrams and continuous-cooling-transformation (CCT) diagrams. TTT diagrams reflect the isothermal decomposition of austenite whereas CCT diagrams provide information under non-isothermal conditions.

An isothermal transformation diagram for a plain carbon steel of eutectoid composition is illustrated in Figure 12 42. The transformation curves for continuous cooling have been added (heavy P_s and P_f lines and shaded area) and four different cooling-rate curves have been superimposed. The Ps lines indicate the point at which pearlite starts to form and the Pf lines the point at which pearlite formation is complete; The Ps and Pf lines are shifted to the right (i.e. longer times) under continuous cooling conditions compared with those for isothermal transformation. The Ms line indicates the start of martensite formation. In order for a fully martensitic structure to be formed the steel must be cooled sufficiently rapidly that the "nose" of the C-curve is avoided. The structures obtained after cooling at the various rates are indicated at the bottom of the diagram. For a steel of hypoeutectoid composition, the isothermal transformation diagrams become slightly more complicated due to the presence of ferrite start (Fs) lines. In the case of more highly alloyed steels, the C-shape of the curves is replaced by more of an S-shaped curve 13. Figure 13.

Hardenability may be defined in terms of an ideal critical diameter D_1 which is the diameter, in inches, of a cylindrical bar that will form 50% martensite at the center during an ideal quench. The 50% martensite value is chosen arbitrarily for convenience in measuring. A bar of a given steel with a diameter greater than the ideal critical diameter cannot be hardened all the way through even by an infinitely rapid

quench. Hardenability is thus an index of the depth to which martensite can be formed in a given steel as the result of a given hardening treatment. In addition to being determined experimentally, the ideal critical diameter may be estimated from the chemical composition of a steel. Two factors are required in order to be able to do this: abase diameter Do that depends on the carbon content and the grain size of the material and the multiplying factors for the various elements present in the alloy. The relationship between base diameter, carbon content and grain size is illustrated in Figure 14 . Multiplying factors for various alloying elements are shown as a function of the amount of the alloying elements in Figure 15 is. From Figures 14 and 15, it may be seen that the ideal critical diameter of a steel with a No. 8 grain size and containing 0.5 w/o carbon, 0.25 w/o manganese, 0.55 w/o molybdenum, and1.8 w/o nickel is:

$Di = Dc \ x \ F_{Mn} \ X \ F_{cr} \ X \ F_{Ni}$ ..... Equation 1

$Df = 0.22 \ x \ 1.8 \ x \ 2.6 \ x \ 1.7 = 1.8$ inches

A similar estimate may be obtained using the hardenability factors given in ASTM method A 255 7.

Using Figure 15, it becomes apparent that some alloying additions are more significant than others in their effect on hardenability. Unfortunately, some of the most potent elements, manganese, chromium, and silicon, have a tendency to form stable oxides. Their use in water atomized prealloyed Low-alloy powders is therefore restricted to Low levels. Manganese is generally kept below 0.35 w/o while chromium and silicon are specified at levels below 0.05 w/o. Fortunately for the powder metallurgist, molybdenum and nickel are good enhancers of hardenability particularly when they are used in conjunction with one another. In fact, for steels containing more than 0.75 w/o nickel, and more than 0.2 w/o molybdenum, the enhanced multiplying factor for molybdenum is shown in Figure 16.

The effect of a small molybdenum addition maybe seen by comparing the isothermal transformation diagram for an AISI 1060 steel with that for AISI 4068 - Figure 17 (a) and (b). The former is a carbon-manganese steel while the latter contains 0.24 w/o molybdenum in addition to the same 0.63 w/o carbon and 0.87 w/o manganese found in the 1060 steel. There is a pronounced shift of the transformation curve to the right in the case of the AISI 4068 steel; from a minimum transformation time of less than 0.5 seconds for the 1060 steel to approximately 3 seconds for the 4068 steel. The time to reach the nose of the curve is greater for the 4068 steel and consequently this steel is more easily transformed to martensite.

Additions of carbon have a lesser effect on hardenability than other alloy additions. The isothermal transformation diagram for an AISI 4047 steel is illustrated in Figure 18 48. When comparing Figures 17 (b) and 18, the additional 0.2 w/o carbon in the 4068 steel does move the curve to the right but only slightly. One significant difference with the change in carbon content is the decrease in the austenite + ferrite region as the carbon content is increased. Even though the contribution of carbon to hardenability is not a major one, the carbon content of the steel affects the maximum attainable hardness of any martensite that is formed - Figure 6.

Development of Sinter-Hardenable Powders

The potential sinter-hardening response of various commercially available powders may be estimated from the Jominy hardenability data for the materials. The recent work carried out in support of the P/M materials standards development program provides a basis for comparing the relative hardenability of different materials °. In order to make a direct comparison between various materials it is important to compare them at the same density because, as was discussed previously, the hardness values obtained during Jominy hardenability testing of P/M materials are a function of density. In Figure 19 (a) and (b) the Jominy distance to an apparent hardness of 65 HRA has been plotted for various materials at a common density of 7.0 g/cm^3. The relatively Low hardenability of the admixed materials is apparent. The diffusion alloyed materials also have what appears to be relatively Low hardenability. However, the heterogeneous microstructure of materials made from diffusion alloyed powders means that apparent hardness measurements for these materials may be somewhat misleading. The hardness value obtained will depend on which microstructural constituent the hardness indenter encounters. The significant difference in the microindentation hardness of various microstructural constituents present in a diffusion alloyed material is illustrated in Figure 20 19 A quantitative estimate of percentage of the various microstructural constituents present in a diffusion alloyed material is a more meaningful test than an apparent hardness measurement.

The prealloyed materials (FL series) shown in Figure 19 (b) with their homogeneous microstructures do not have particularly high hardenability. However, the addition of copper to the nickel-molybdenum prealloy (FL-4605) enhances the hardenability considerably as seen in the result for the FLC-4608 material. In fact, sinter-hardened parts have been made for many years by sintering FLC-4608 materials in conventional sintering furnaces. The hardenability multiplying factor for copper, according to Grossmann, is shown in Figure 21 20. There is, however, a need for materials with greater hardenability than those that are currently available so that larger P/M parts may be sinter-hardened. In addition, while sinter-hardened parts made from FLC-4608 materials may have good apparent hardness they do not always possess good tensile and fatigue properties.

What are the requirements for a good sinter-hardenable alloy?

- Good hardenability - better than the hardenability of FLC-4608,
- Good compressibility - greater than 6.8 g/cm3 at 40 tsi compaction pressure, and
- Compatibility with sintering at 2050 F (1120 C).

The focus of alloy development should be to utilize alloying additions in combinations that optimize the synergistic effect of their presence on the hardenability of the alloy. For P/M steels this would involve various combinations of alloying elements such as nickel, molybdenum, manganese, and chromium. Manganese and chromium have a significant influence on hardenability but their use is restricted because of their strong affinity for oxygen. While molybdenum and nickel additions cost more than additions of manganese and chromium their oxides are reducible during powder annealing and nickel-molybdenum prealloyed P/M steel powders may be sintered at 2050°F (1120°C).

While the contribution of various elements with respect to hardenability is important, the amount of each alloying addition needs to be a compromise between hardenability enhancement and reduced compressibility of the prealloyed powder. Elements that have a strong solution strengthening effect in ferrite reduce powder compressibility; nickel and manganese fall into this category. The substitution solution strengthening effect of molybdenum and chromium is less than that of nickel and manganese - Figure 22 21,22. Elements that form interstitial solutions with iron, such as carbon and nitrogen, need to be kept to very Low levels in order to produce compressible powders. A recent experimental program, focused on sinter-hardenable materials, has resulted in the development of new powders 23. One of the powders is a highly hardenable P/M steel that, when mixed with 2 w/o copper powder and 0.9 w/o graphite, reaches a green density in excess of 6.8 g/cm^3 at a compaction pressure of 40 tsi and develops a sintered microstructure with 90% martensite at the core of 1.25 inch diameter parts with a mass of over 350 g. An ultimate tensile strength of 120,000 psi (825 MPa), 0.2% offset yield strength of 99,000 psi (680 MPa), and elongation of 1% was obtained using fiat unmachined tensile bars compacted at 40 tsi. The test bars had a sintered density of 6.9 g/cm^3 and an apparent hardness of 32 HRC after tempering at 400°F (200o0) in air for one hour. The use of machined round bars, the type normally used for heat treated *PIM* materials, would increase both the tensile strength and the ductility values obtained. Another powder from the study has higher compressibility and somewhat less hardenability but still results in parts with an apparent hardness of over 30 HRC in parts with smaller cross-sections.

Advantages of Sinter-Hardening

There are a number of benefits of the sinter-hardening process compared with liquid quench-hardening of *PIM* materials.

- The need for a secondary quench-hardening treatment is eliminated
- The reduced distortion of parts due to the less severe quench leads to better dimensional control
- The tempering of sinter-hardened parts in air is easier than for quench-hardened parts. Parts that have been quenched in an oil bath retain a considerable amount of oil in their pores. If tempering at a temperature above 400°F (200°C) is required the oil quenched parts must first be tempered below 400°F (200°C) to burn off the entrapped oil prior to tempering at the higher temperature.
- Sinter-hardened parts do not need an oil removal step prior to finishing operations such as   plating.

While it is possible to perform limited machining on sinter-hardened parts, they will, for the most part, be designed as net-shape parts. For some applications, the combination of warm compaction with sinter-hardening may be used to overcome the shape limitations of rigid die compaction. Features may be machined while compacts are in the "green" state prior to sinter-hardening. Typically, sinter-hardened parts need tempering to relieve stresses and improve toughness. The high apparent hardness of sinter-hardened parts makes sizing difficult if not impractical.

The application of sinter-hardening is expected to increase significantly over the next few years. New, highly hardenable, powders will be developed and furnace makers will

enhance the systems they have designed for the accelerated cooling of sintered parts.

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Figure 1: As-sintered microstructure of FN-0205.

0.2 w/o
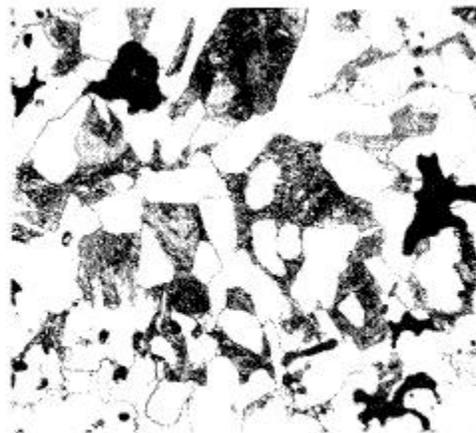
0.4 w/o

0.6 w/o

0.8 w/o

Figure 2: As-sintered Microstructure of Carbon Steels with Various Combined Carbon Contents.

% ferrite   = {(0.77 - 0.40)/(0.77 - 0.02)} x 100 % = 49.33 %
% austenite = {(0.40 - 0.02)/(0.77 - 0.02)} x 100 % = 50.67 %
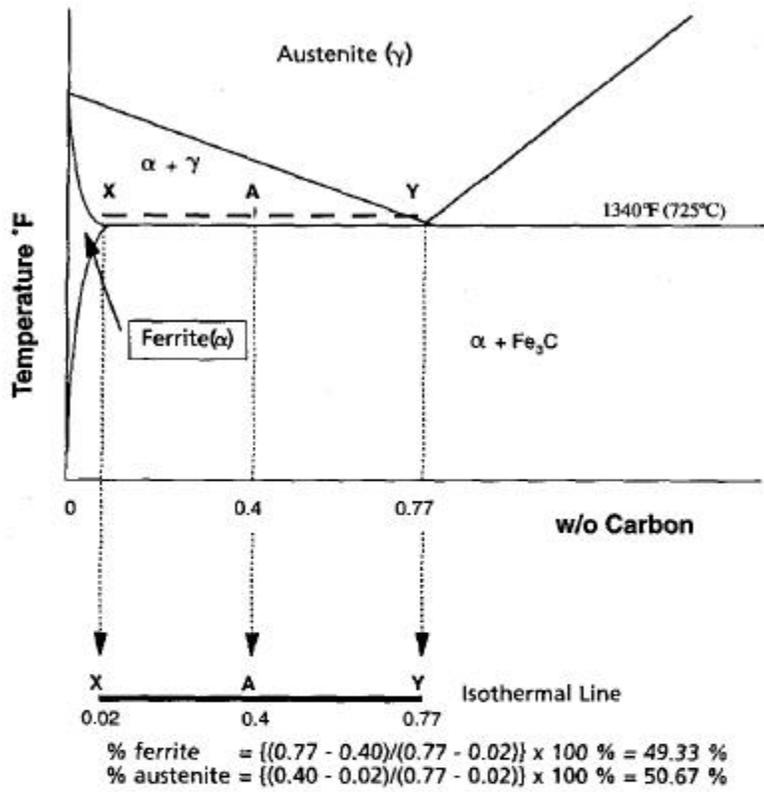
Figure 3: The Use of the "Lever Rule" to Determine the Relative Amounts of Ferrite and Pearlite Expected in a Hypoeutectoid Steel.
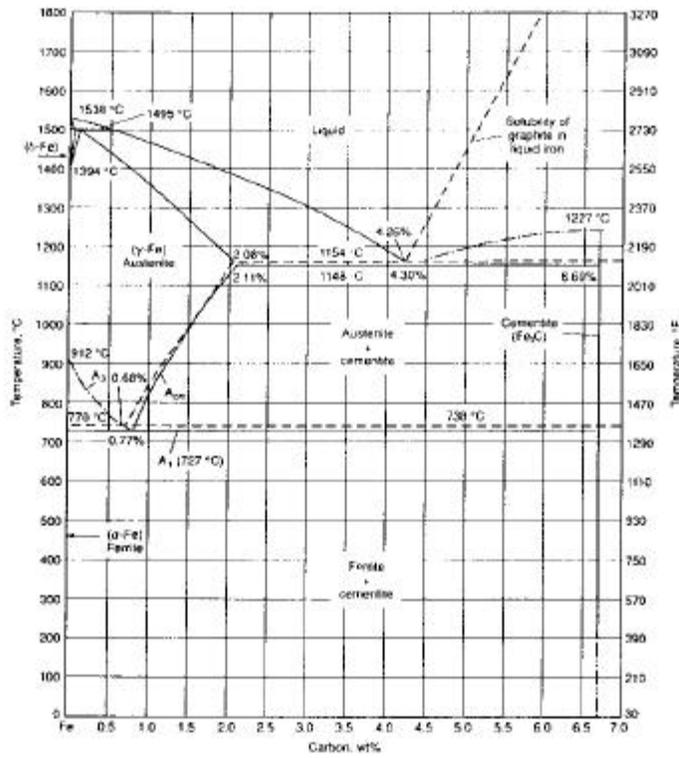
Figure 4: Carbon-Iron Equilibrium Phase Diagram (Reference 1)
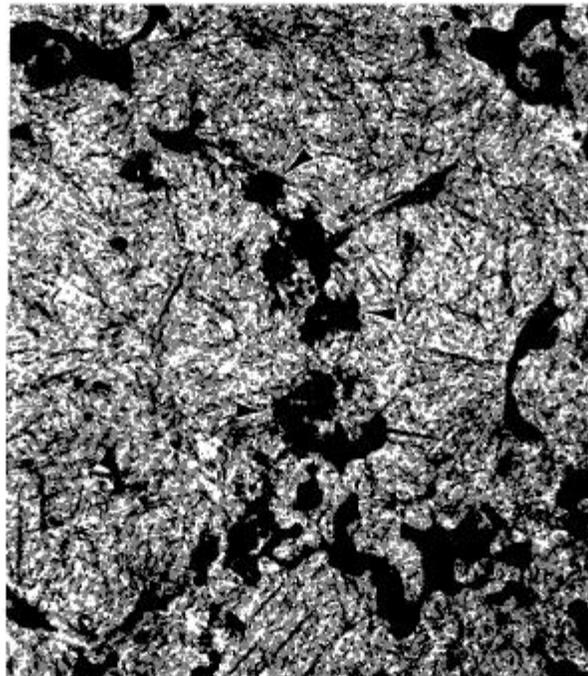
Figure 5: Tempered Martensite in Quench-Hardened and Tempered F-0008.
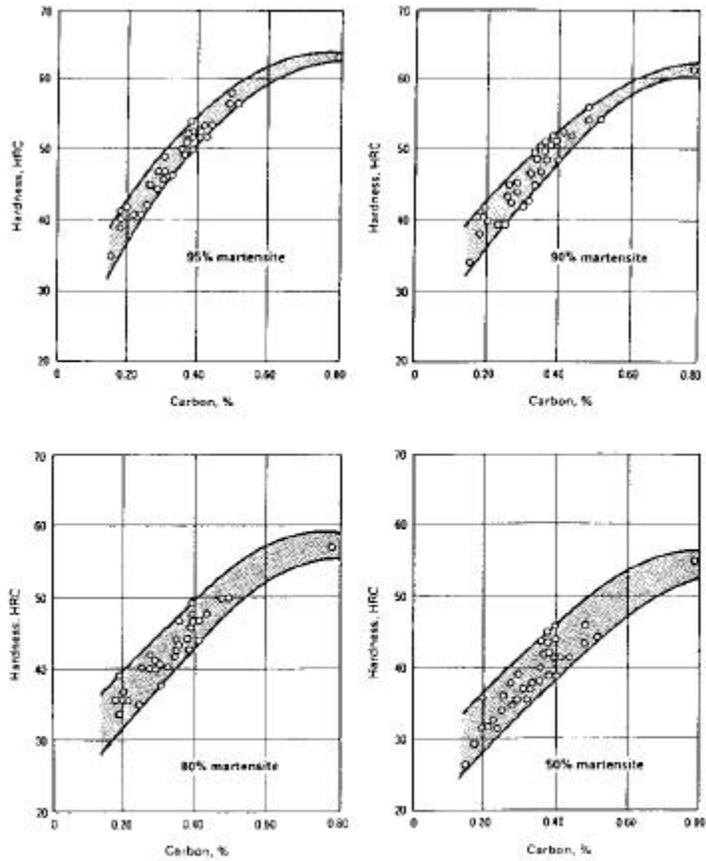
Figure 6: Effect of Carbon Content on the Hardness of Martensitic Structures (Reference 6).

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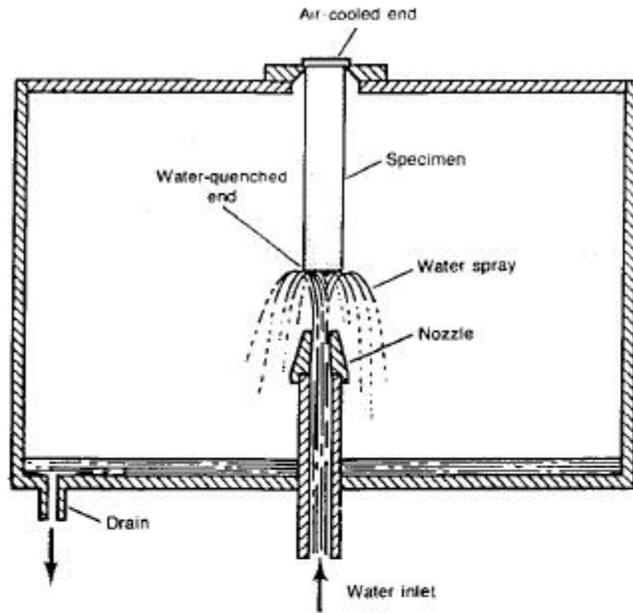
Figure 7: Jominy End-Quench Apparatus (Reference 6).
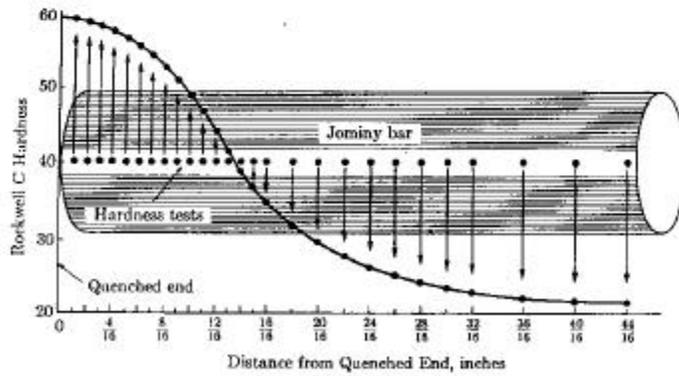
Figure 8: Jominy Curve Obtained by Plotting the Hardness Values Measured Along the Length of the Quenched Bar (Reference 8).
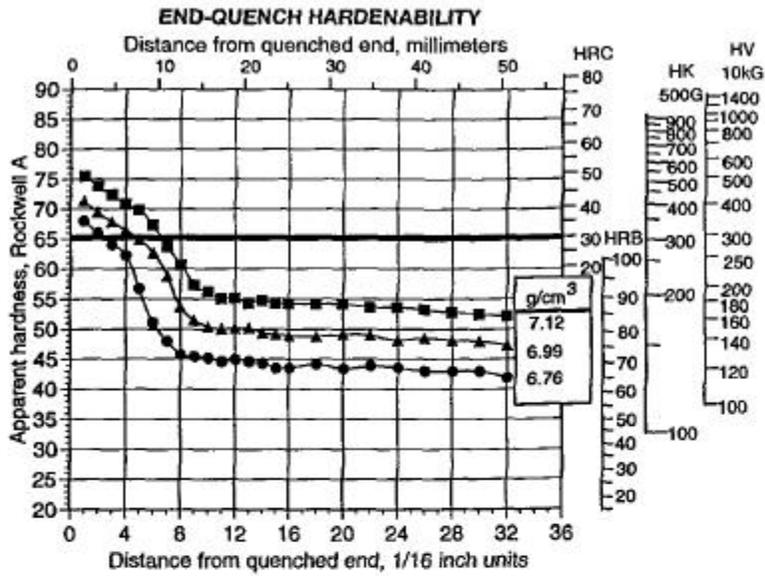
Figure 9: Effect of Density on the Jominy Hardenability of FL-4605 (Reference 9).
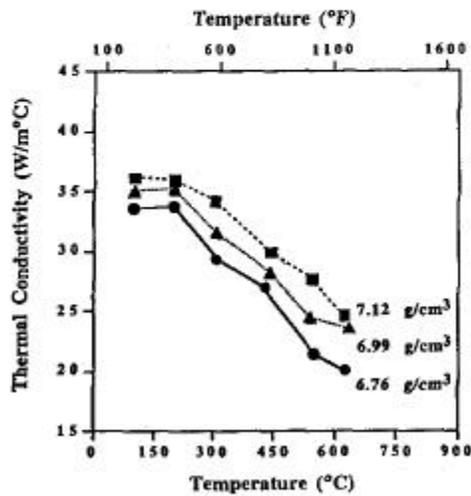
Figure 10: Effect of Density on the Thermal Conductivity of FL-4605 (Reference 9).

17
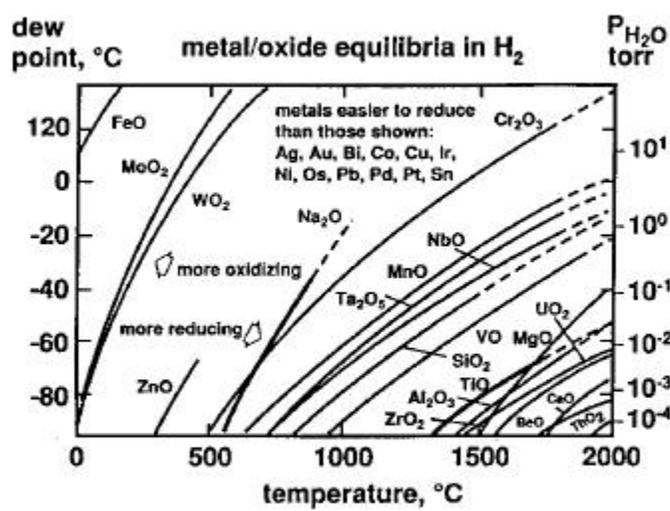
Figure 11: Metal/Oxide Equilibria in Hydrogen (Reference 11).
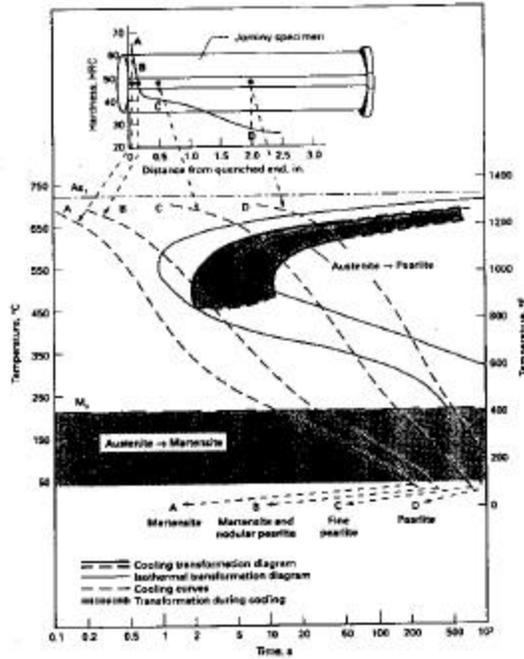
Figure 12: Relationship Between CCT (heavy lines) and TTT (light lines) Diagrams of Eutectoid Steel. Four Cooling Rates from Different Positions on a Jominy End-Quench Bar are Superimposed on the CCT Diagram (Reference 12).

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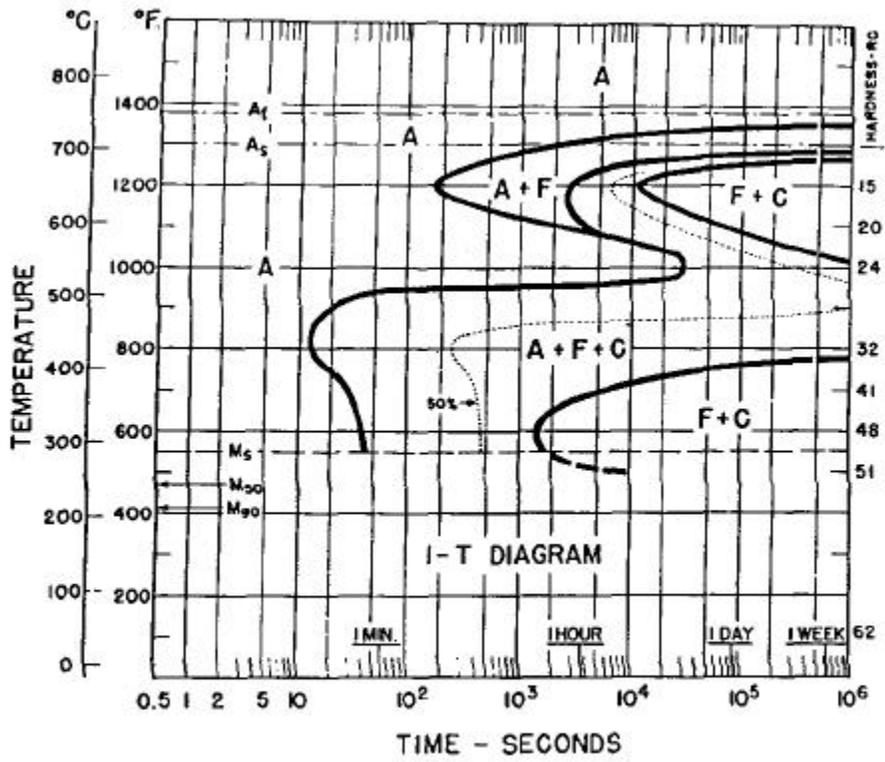
Figure 13: TTT Diagram for 4340 Steel (Reference 13).
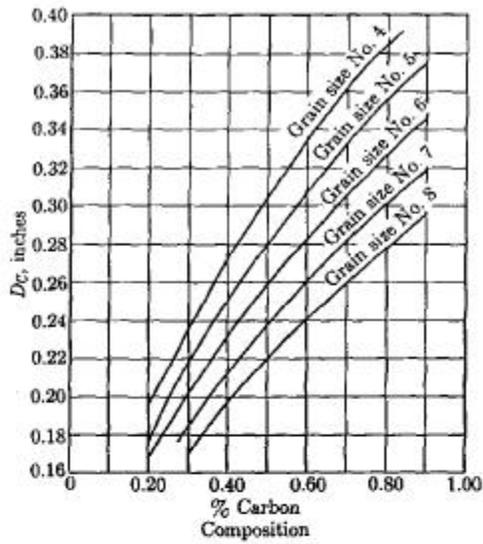
Figure 14: Effect of Carbon Content and Grain Size on the Base Diameter used in Calculating the Idael Diameter of a Steel (Reference 14).
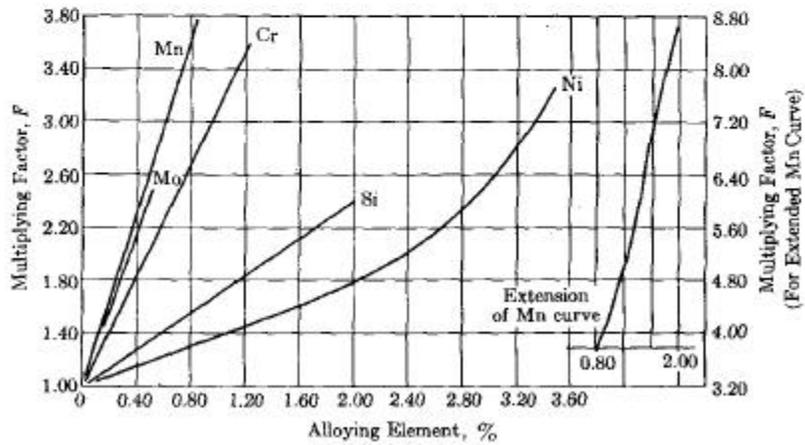
Figure 15: Multiplying Factors for Several Common Alloying Elements (Reference 15).

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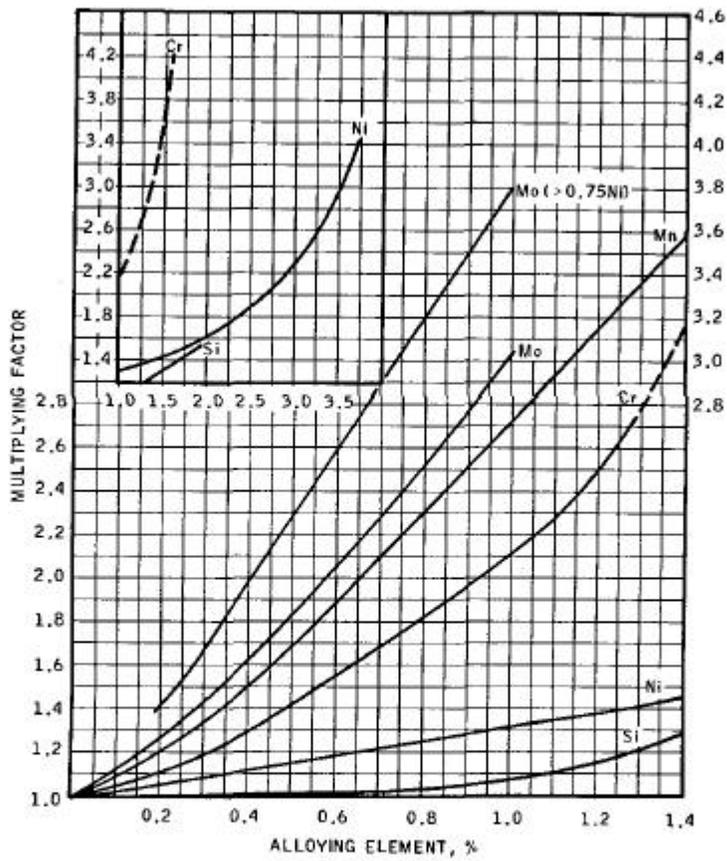
Figure 16: Average Multiplying Factors for Several Alloying Elements (Reference 16).
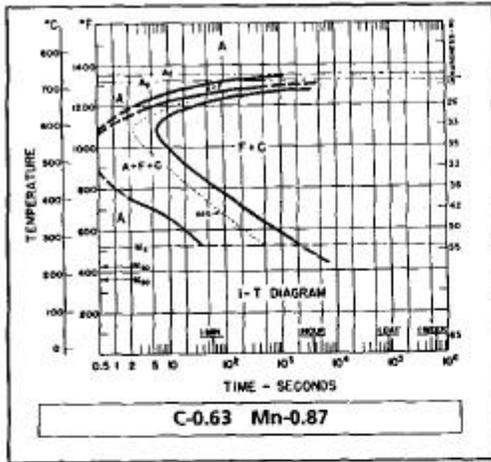
(a)
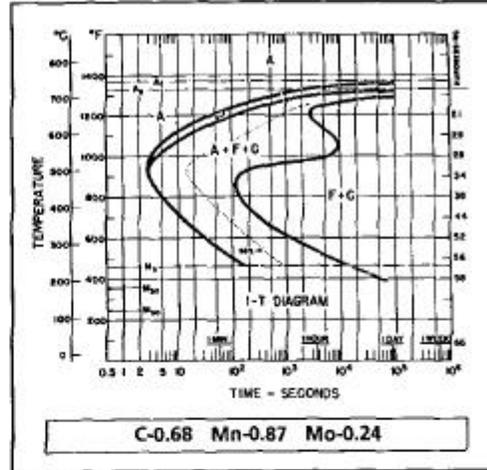
(b)

Figure 17: Isothermal Transformation Diagrams: (a) 1060 (b) 4068 Steel (Reference 17).
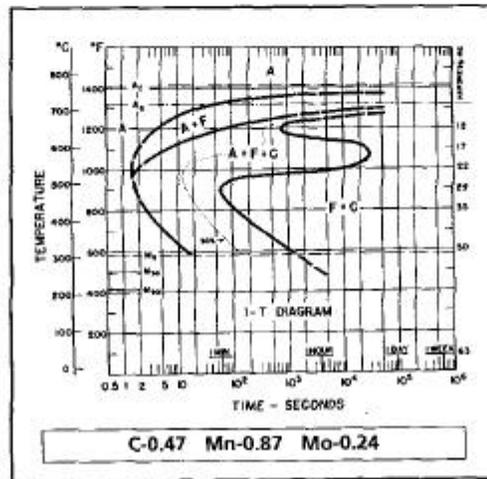
Figure 18: Isothermal Transformation Diagram for 4047 Steel (Reference 18).
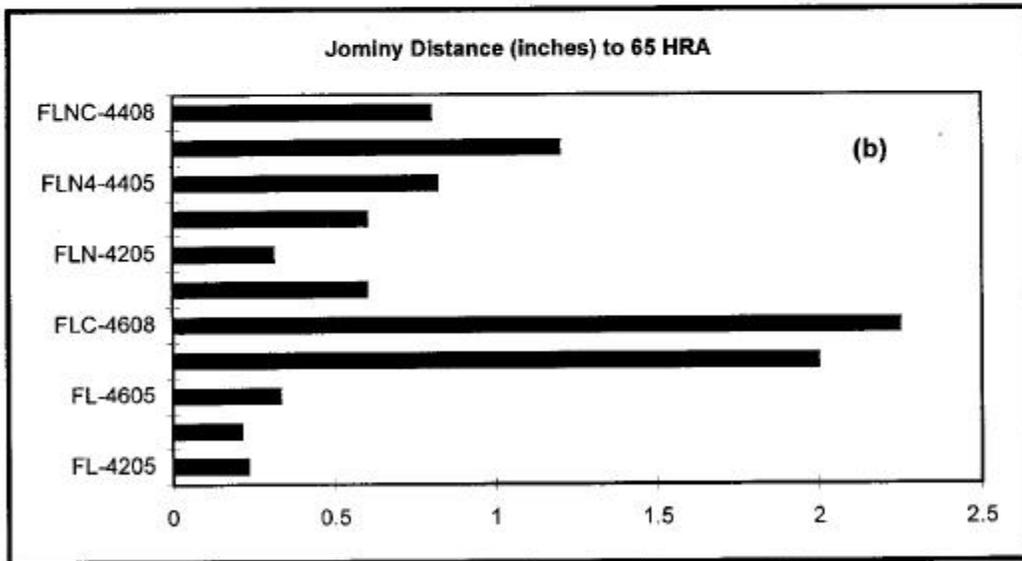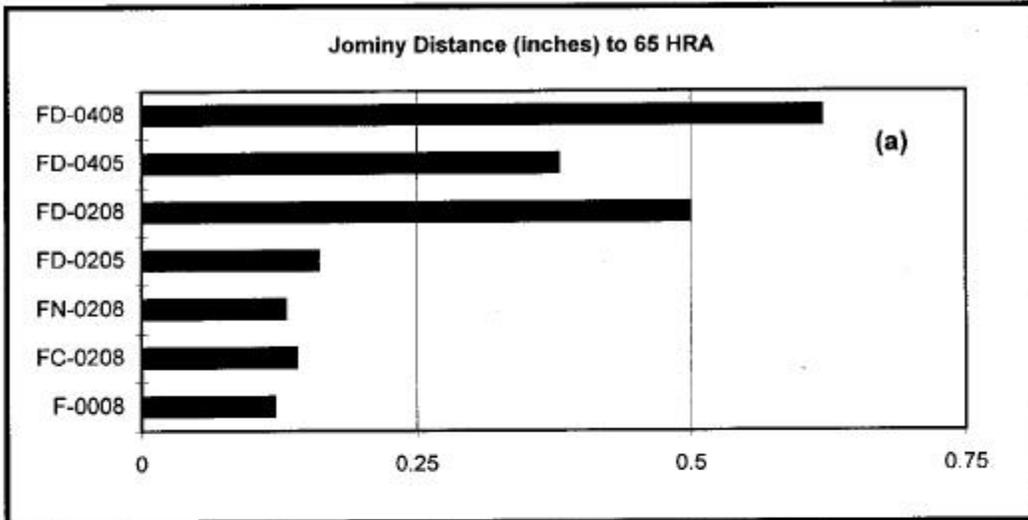
Figure 19: Jominy Distance to an Apparent Hardness of 65 HRA for Various P/M Materials at a Density of 7.0 g/cm³ (Data from Reference 9).

MICROHARDNESS (HV$_{20}$)				
P	F$_{nr}$	NR	DM	LM
262	287	139	635	442
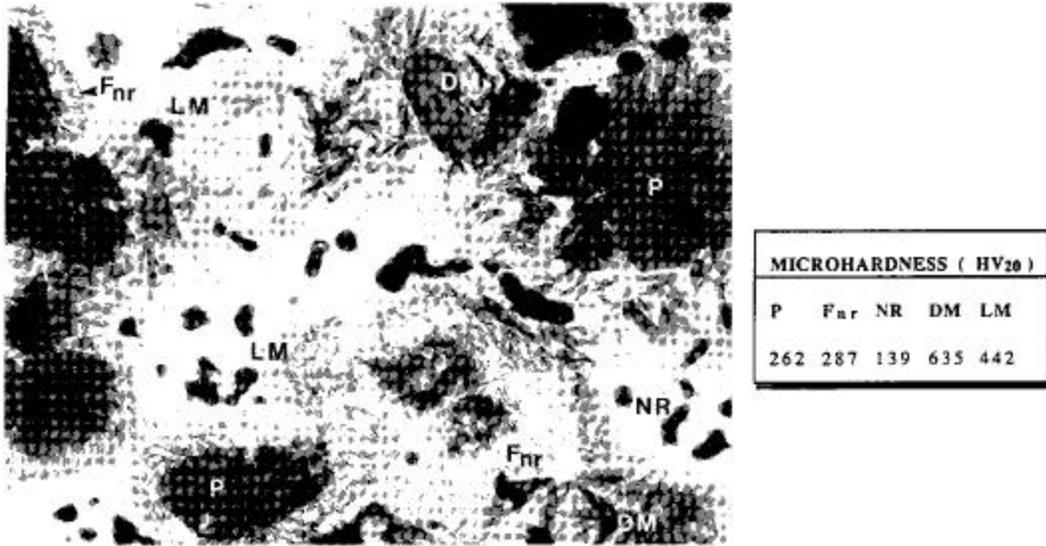
Figure 20: Microindentation Hardness of Various Microstructural Constituents of Sinter-Hardened FD-0408 + 2 w/o Nickel (Reference 19).
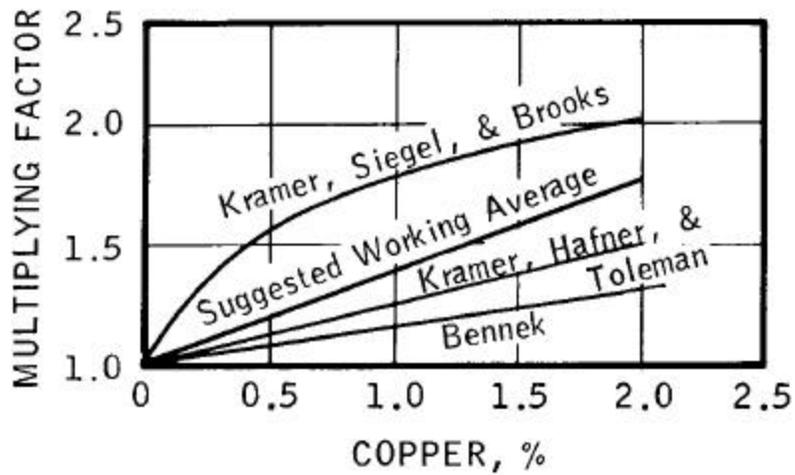
Figure 21: Multiplying Factors for Calculating the Effect of Copper on Hardenability (Reference 20).