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Application of Phase-Contrast Metallography In a Production Laboratory

Metallographic examination is an important tool for the control of heat treating of metals and alloys. The primary function of heat treating is to change some properties of metals and alloys favorably by altering the crystal and grain structures. Precise determination of the internal structure is an important guide for the control of the various treatments.

Proper service of modern ultra-high-speed machines depends directly on the reliability of all components and requires the best possible properties, a high degree of homogeneity and uniformity, close tolerances and low-cost production methods.

The majority of mechanical components in IBM machines are heat treated. The most important of these treatments is case hardening. This treatment is applied to steel parts mainly by gas carburizing and carbonitriding. These processes have proven the most adequate for economic precision treatment of small, intricate components. Some of these parts are as thin as 0.015 inch and have a case-depth requirement of 0.001-0.002 inch with a specified peak hardness of Rockwell C 62. The material used to avoid warpage is often alloy steel (such as AISI 8620). This requires careful control of hardening temperature and quenching speed. Slight deviations may result in either a through-hardened section or a case softer than required. Minor changes in temperature, atmosphere, time and speed produce a great variety of microconstituents both in case and core. The identification of these is sometimes difficult. Some of the microconstituents found in case-hardened components are: ferrite, lamellar pearlite (in grain sizes ranging from coarse to extremely fine), cementite and other carbides, upper and lower bainite, tetragonal and cubic martensite, and tempered martensite.

The exact determination of the constituents in the structure of a case or core is important in controlling heat-treating operations and maintaining required close tolerances and properties. The commonly used microscopic methods, however, are not always adequate to differentiate closely related microconstituents such as cubic martensite and lower bainite, or upper bainite and fine pearlite. These microconstituents frequently cause undesirable effects, e.g., uneven or generally poor wear resistance, low impact strength and premature fatigue failure.

Phase-contrast microscopy is a newly added feature in modern metallography which has proved useful in identifying some microconstituents of inhomogeneous areas in case-hardened steel components. This note presents a summary of the method and characteristic problems solved with the aid of phase-contrast microscopy. The application to *opaque specimens* is discussed, whereas most of the literature on the phase-contrast method concerns transparent specimens.

Although the various microconstituents in steel are rather well known and understood by metallurgists, there is still a controversy of opinions and theories about the so-called "intermediate transformation structures." X-ray diffraction is the proper tool for precise determination of crystal structures, but its applications are limited and the method is too time-consuming for practical use in a production laboratory. The conventional metallographic microscope continues to be the most adequate general instrument for examination of heat-treated components. In conjunction with other equipment, such as a microhardness tester, it solves most problems. Its limitation, however, is that it merely gives a highly magnified image for the human eye.

The polished and etched metallographic specimen has a definite surface pattern conforming to the grain structure. This pattern changes the intensity of reflected light or promotes a selective reflection of certain wavelengths. Light reflected from a metallographic specimen may, however, suffer alteration in phase from small local vertical displacement between slightly different structural elements or from variances of surface optical activity. This does not contribute to the efficiency of the examination, as the human eye cannot distinguish between light rays differing in phase. The phase-contrast microscope senses the small differences in phase and transforms them into observable modulations of light intensity. Structural details that otherwise would be unobserved may thus be seen.

Ordinary metallographic microscopes create the final image with the undeviated direct light reflected from the specimen and all diffracted orders still collected by the objective. The diffracted light, recombined with the undeviated light, produces a modulation of light intensity which resembles the specimen structure. The phase-contrast microscope makes observable the phase differ-

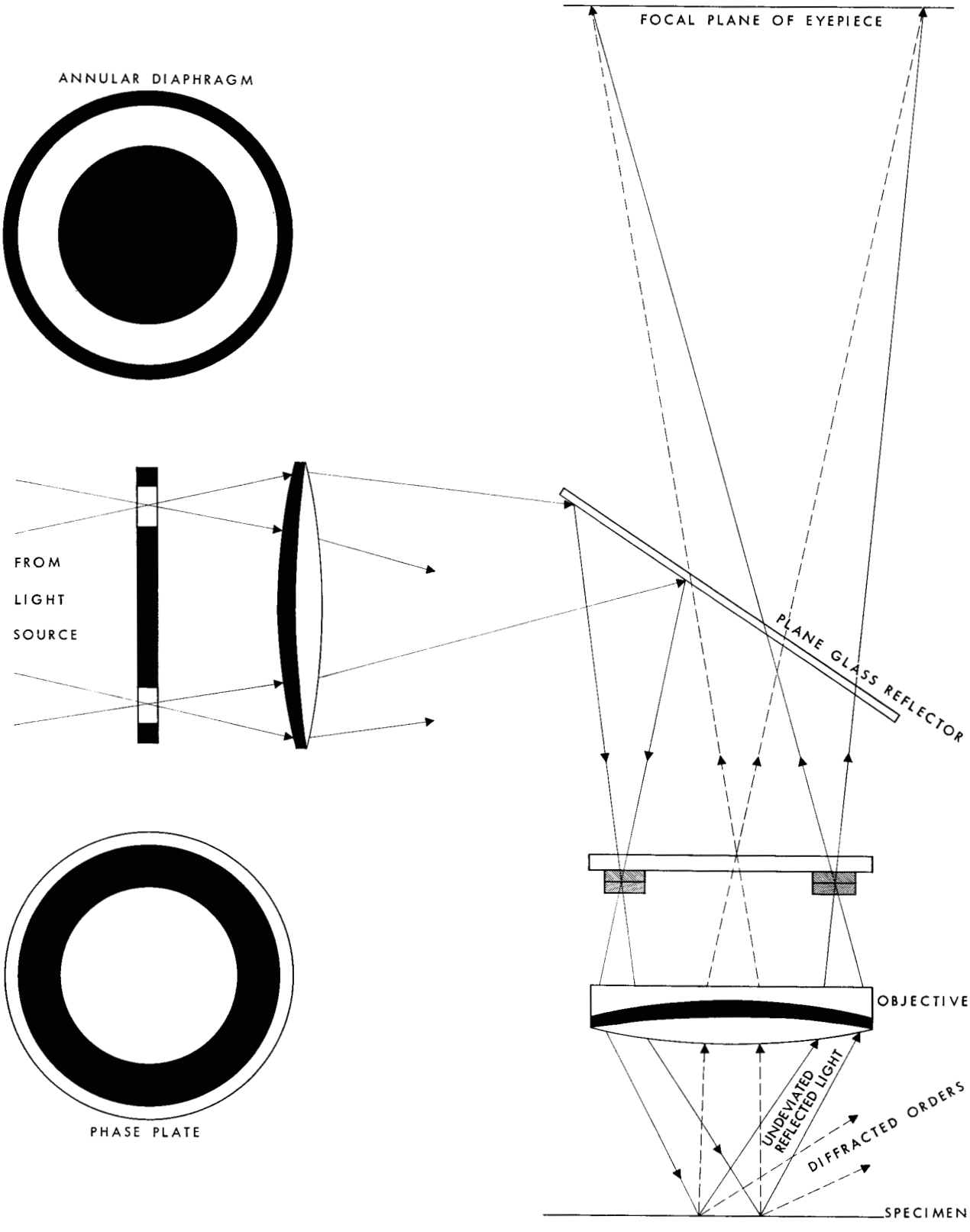

Figure 1 **Schematic principle of phase-contrast illumination.**
(Adapted from Ref. 1, p. 8. Courtesy of the American Society for Metals.)

ences of diffracted orders, thus improving the contrast of the final image.

Figure 1 illustrates the optical principles of the phase-contrast metallurgical microscope. This is a conversion of the ordinary bright-field illumination by the addition of an annular diaphragm, where the image of the light is formed. The annular diaphragm is thus imaged in the rear focal plane of the objective. The objective, in turn, will create displaced images of the annular diaphragm by the undeviated, directly reflected light and diffracted orders. When a phase-retarding plate the same size as the annular diaphragm is inserted into the rear focal plane of the objective, either the undeviated light or the diffracted orders can be altered in phase with respect to the other. At the same time a proper material in the phase-retarding plate (evaporated coating of inconel or aluminum) reduces intensity of the undeviated light and increases intensity of the diffracted light.

The metallographic microscope used for the examinations was the American Optical #2400 P Research Model. The phase-contrast equipment of this unit differs only slightly from the general principle described above. The phase-retarding plate is removed from the rear focal plane of the objective and placed at another point in the optical system. This change does not alter the basic principles of the method.

No additional sample preparation is required for phase contrast beyond that necessary for bright-field illumination. All specimens were polished mechanically and etched with two percent nitric acid alcoholic solution for approximately five seconds. Three photomicrographs were taken for each specimen. The first photomicrograph in each sequence shows a general view of the selected area in a moderate magnification ($\times 200$) with conventional bright-field (carbon-arc) illumination and a green filter. The second and third photos show a characteristic portion of inhomogeneous structure in a magnification of $\times 1000$ (with dry objective) and a light-blue filter. Bright-field illumination was used on the second photomicrograph and phase contrast was used on the third. A comparison between the photomicrographs demonstrates the capabilities of phase-contrast metallography. The Kodak metallographic plates were developed in Kodak DK 60 standard solution for five minutes and printed on Kodak AZO paper.

From a stock of five hundred specimens, eighteen characteristic samples based on reports and notes of investigations were selected. After a careful examination of the structures, preliminary exposures were taken from typical areas. After evaluation of the preliminary photomicrographs, four representative specimens were chosen for publication. These photomicrographs (Figures 2 to 13) follow on pages 88-91.

Figures 2 to 4 show microstructures of a roller stud, the first specimen selected (*A*). Photomicrograph *A*1 in Figure 2 shows the general area of investigation. The spot designated in the next two pictures (enclosed in a rectangle) is the inner part of the case, which was softer than desirable. The Knoop hardness (measured with a 500-gram load) of this area was 580-670. This is equivalent to Rockwell C 52-57. The material was AISI C 1213 steel. (Nominal composition: C = 0.13 max; Mn = 0.70/1.00; P = 0.07/0.12; S = 0.24/0.33.)

Figure 3 (photomicrograph *A*2) is the same area as Figure 2 with ordinary bright-field illumination. The structure is extremely heterogeneous and microconstituents are not clearly visible. Some martensite needles can be identified, but the matrix structure can only be assumed to be bainite.

Figure 4 (photomicrograph *A*3) shows the same spot in the same magnification as *A*2, but with phase-contrast illumination. A comparison of *A*3 and *A*2 indicates that:

1. The phase-contrast illumination shows more martensite (see arrows in *A*3).
2. The matrix structure is almost uniform and is identified as lower bainite. The different layers in the bainitic matrix may be recognized as diffusionless transformation products of iron and precipitated carbide plates.

Figures 5 to 7 show the core structure of a stud made of AISI 1213 grade steel. The banded structure shown in the general view picture *B*1 (Figure 5) is extremely inhomogeneous and the hardness was uneven, varying between Knoop 310 and 195. The two high-magnification photomicrographs, *B*2 and *B*3 in Figures 6 and 7, are typical representations of a case where neither technique adequately reveals all structural details. The two methods, however, offer a perfect solution if considered together.

In the conventional bright-field illumination three microconstituents can easily be identified: (1) the lower bainite plates with typical triangular pattern in the center section, (2) the free ferrite grains, (3) the manganese-sulfide inclusions. There is no clear indication of the matrix structure. The phase-contrast micrograph *B*3 (Figure 7) indicates also the lower bainite plates, but does not show the correct amount of free ferrite. Nevertheless, it can be observed in this picture that the matrix structure is partly upper bainite (such as in the lower right-hand corner of the micrograph) and partly fine pearlite. This structure is responsible for the great deviations in hardness.

Figures 8 to 10 show the center section of a tooth in a gear. Micrograph *C*1 (Figure 8) presents again the general view of the area. The rectangle indicates the location of the detailed examination. A portion of the hardened case with part of a microhardness traverse may be observed in the lower left-hand corner. Hardness of the structure shown in micrographs *C*2 and *C*3 (Figures 9 and 10, respectively) was Knoop 410-460, which is equivalent to Rockwell C 41-45. The material is AISI C 1117 grade steel. (Nominal composition: C = 0.14/0.20; Mn = 1.00/1.30; P = 0.040 max; S = 0.08/0.13.)

This is an unusual example in which phase-contrast microscopy offers a complete answer to the question of microstructure, even without support of conventional microscopy. Standard bright-field illumination in micrograph *C*2 (Figure 9) shows some bainite plates but does

(Text continued on page 92)

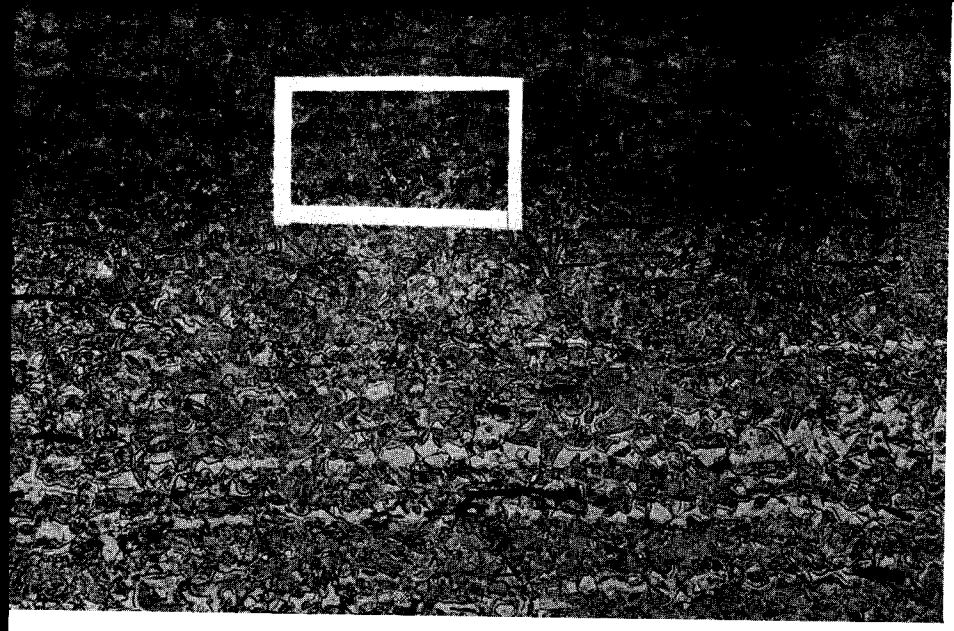

Figure 2
General view of the area examined in specimen *A*. Rectangle shows area of photomicrographs *A2* and *A3* (Figures 3 and 4).

A1
Magnification ×200
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Figure 3
Inhomogeneous structure of sample *A* in bright-field illumination.

A2
Magnification ×1000
Reduced 25% in reproduction

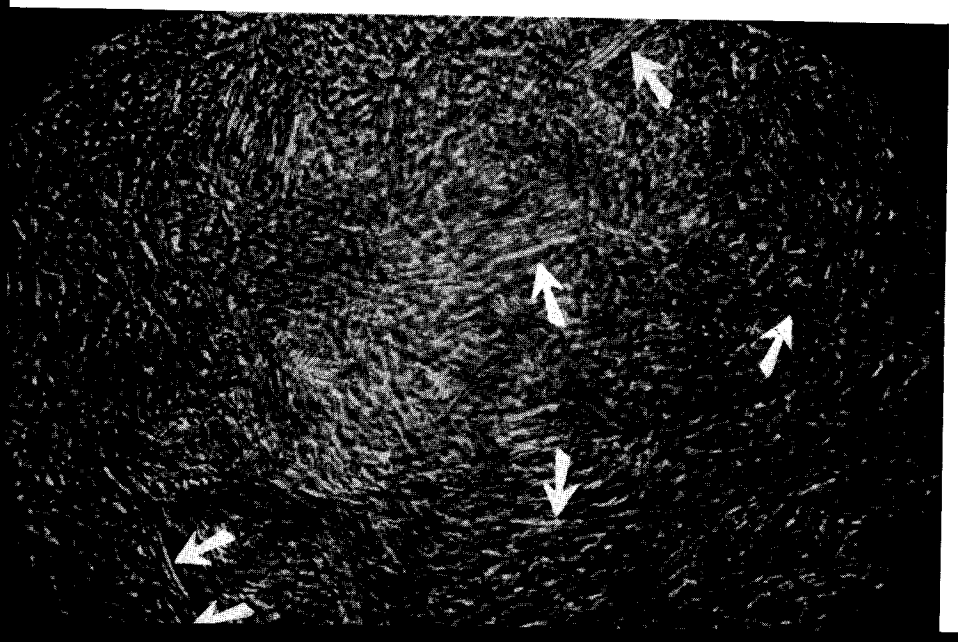

Figure 4
Same area as in *A2* (Figure 3) with phase-contrast illumination. Arrows point to martensite needles.

A3
Magnification ×1000
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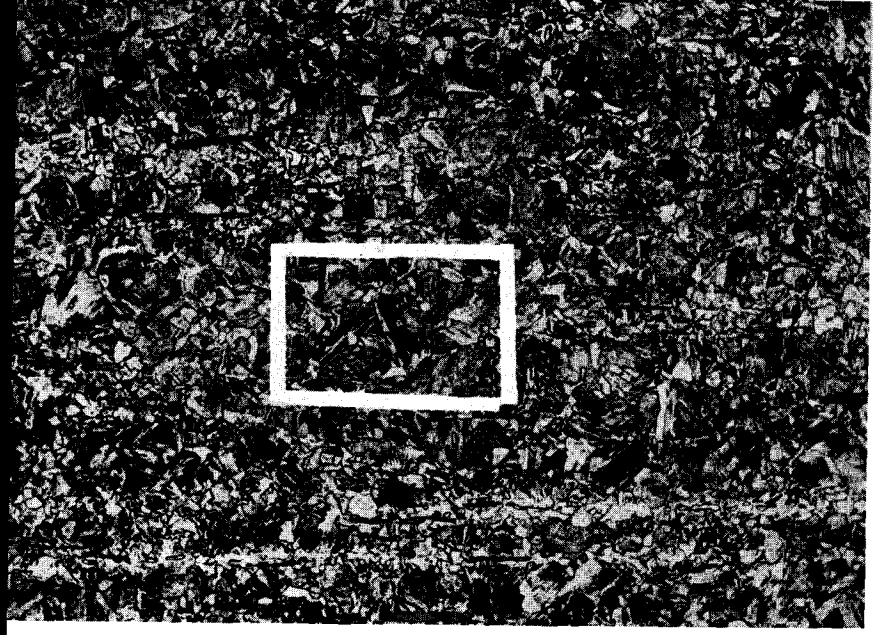

Figure 5
General core structure of specimen *B*. Area of detailed examination shown within rectangle.

B1
Magnification ×200
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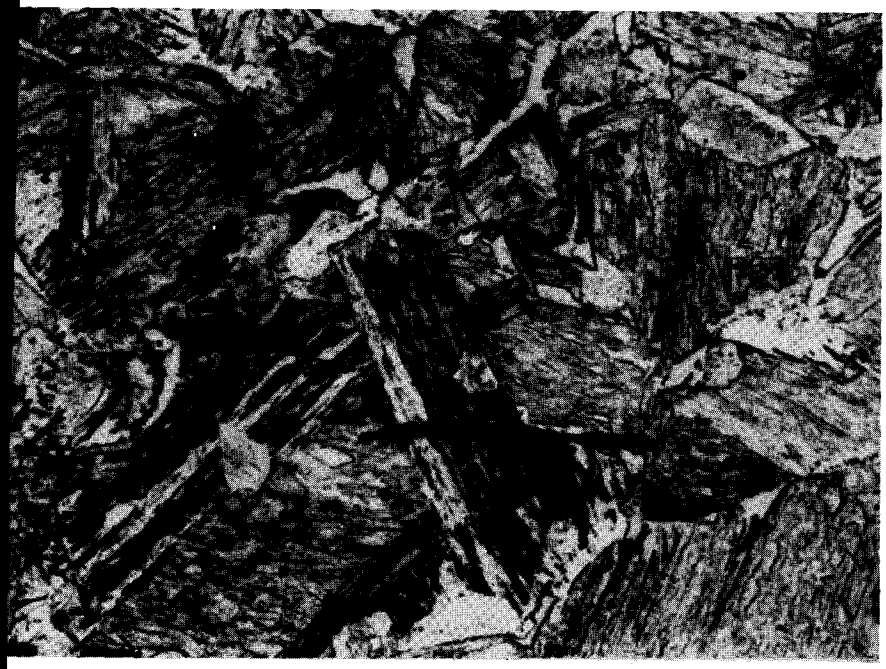

Figure 6
Bright-field illumination: small, marked area of B1 (Figure 5).

B2
Magnification ×1000
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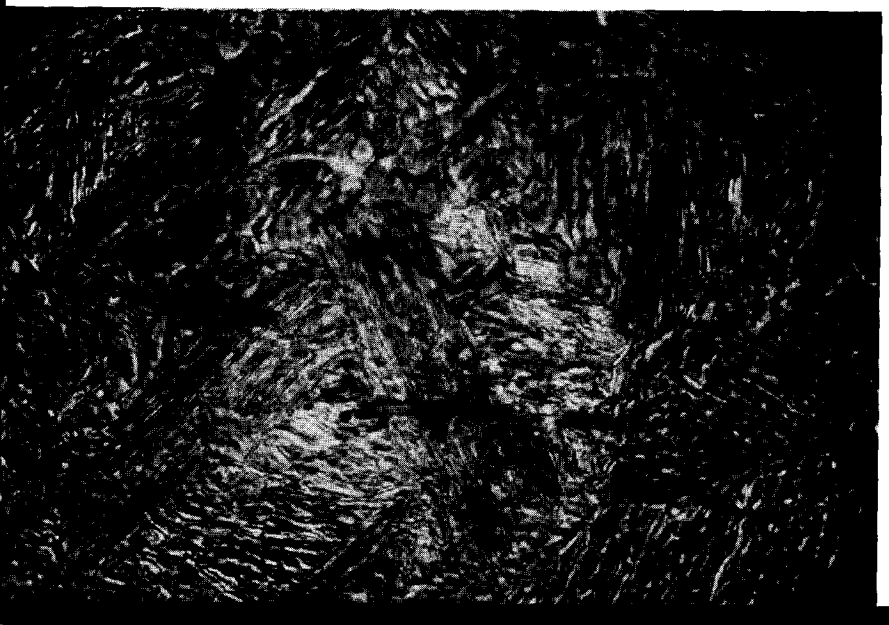

Figure 7
Phase-contrast: same area as shown in B2 (Figure 6).

B3
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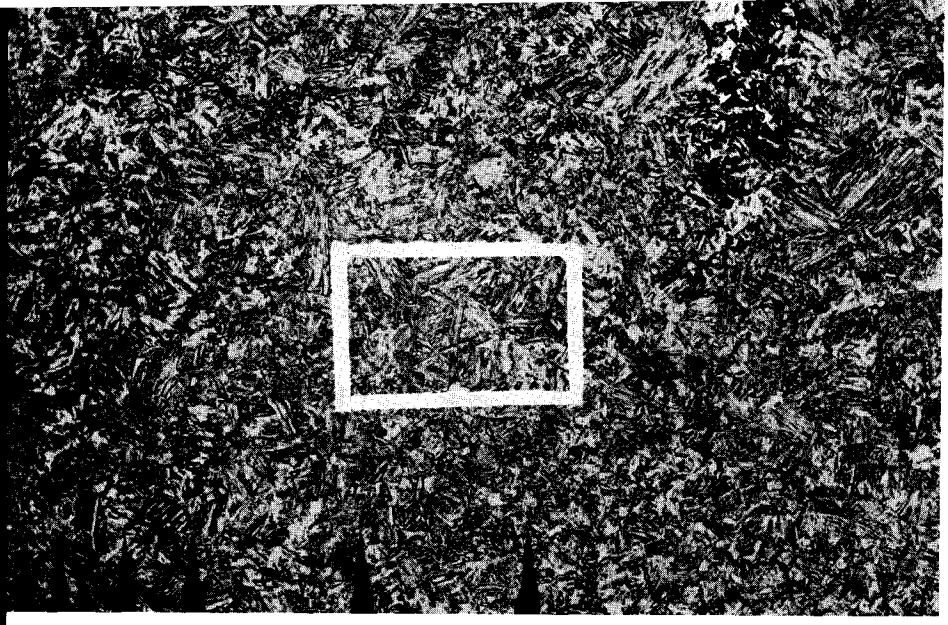

Figure 8
General view of structure at center of tooth of specimen C with the area of detailed examination marked with rectangle.

C1
Magnification ×200
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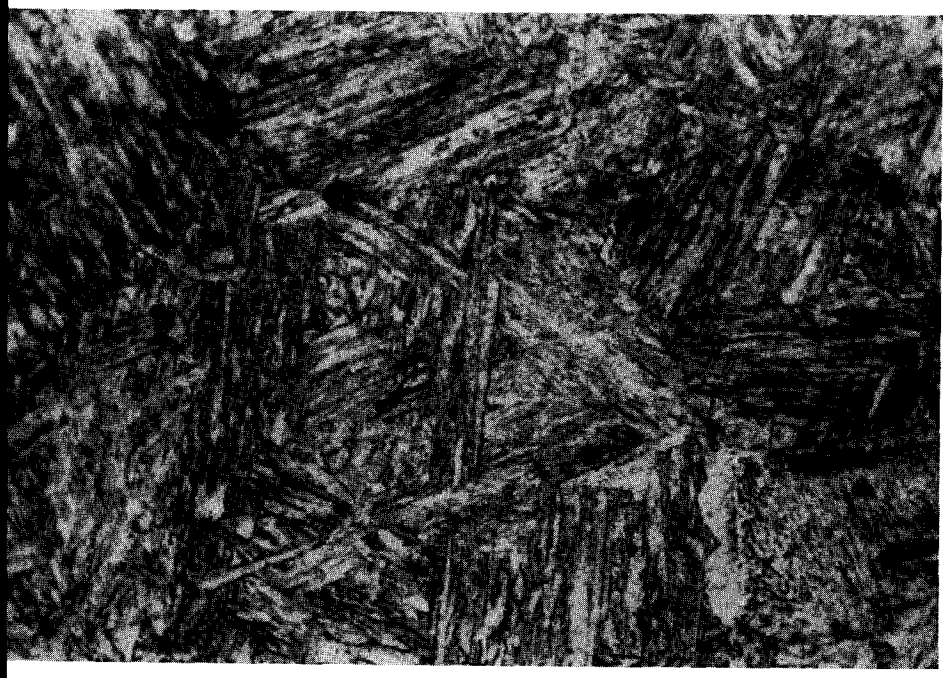

Figure 9
Small area in center of C1 (Figure 8) magnified with conventional bright-field illumination.

C2
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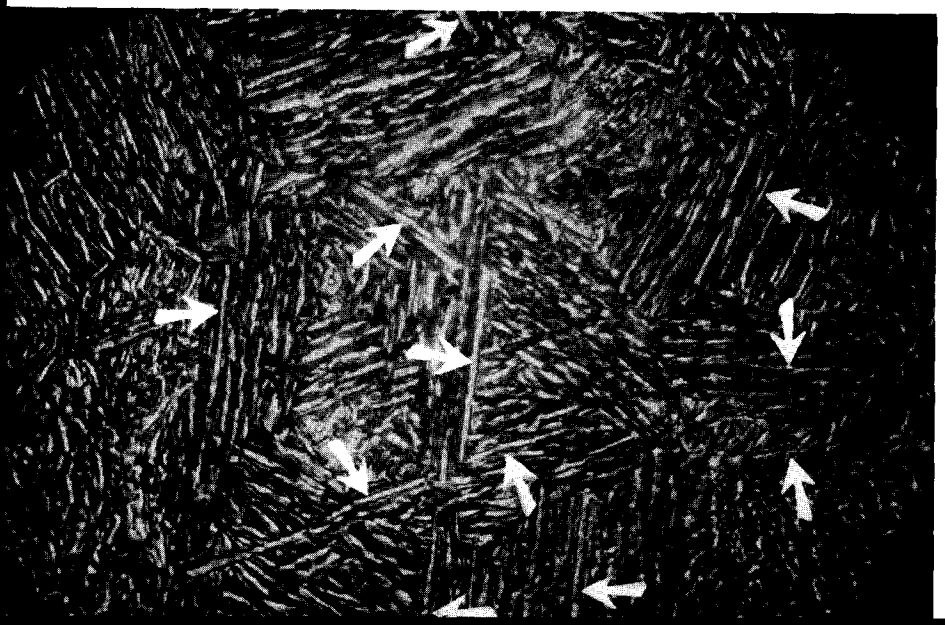

Figure 10
Same area as in C2 (Figure 9) with phase-contrast illumination. Lower bainite plates marked with arrows.

C3
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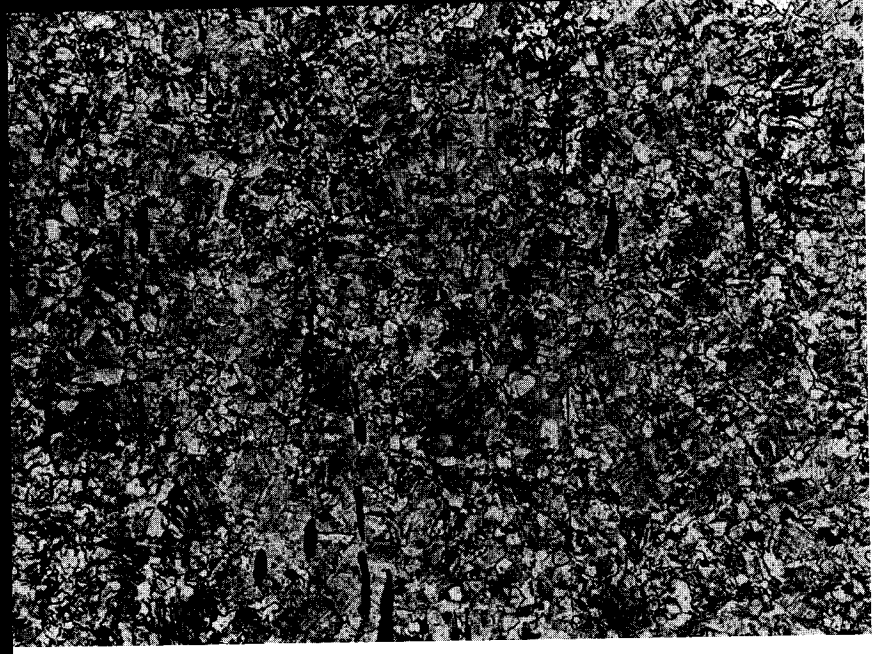

Figure 11
General view of structure of specimen *D* around the area of pictures *D2* and *D3* (Figures 12 and 13).

D1
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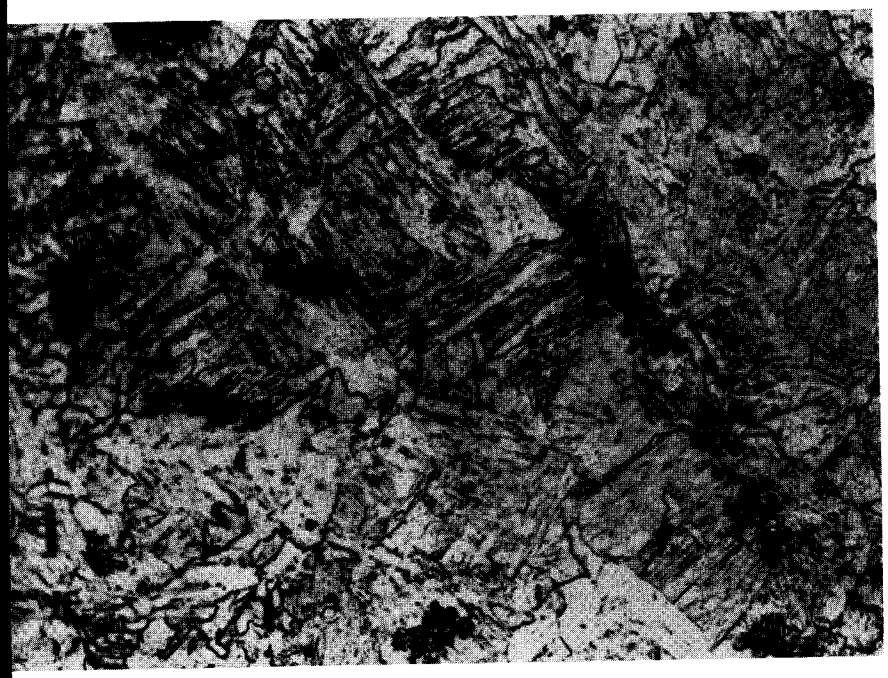

Figure 12
Structure near the center of picture *D1* (Figure 11) with bright-field illumination.

D2
Magnification ×1000
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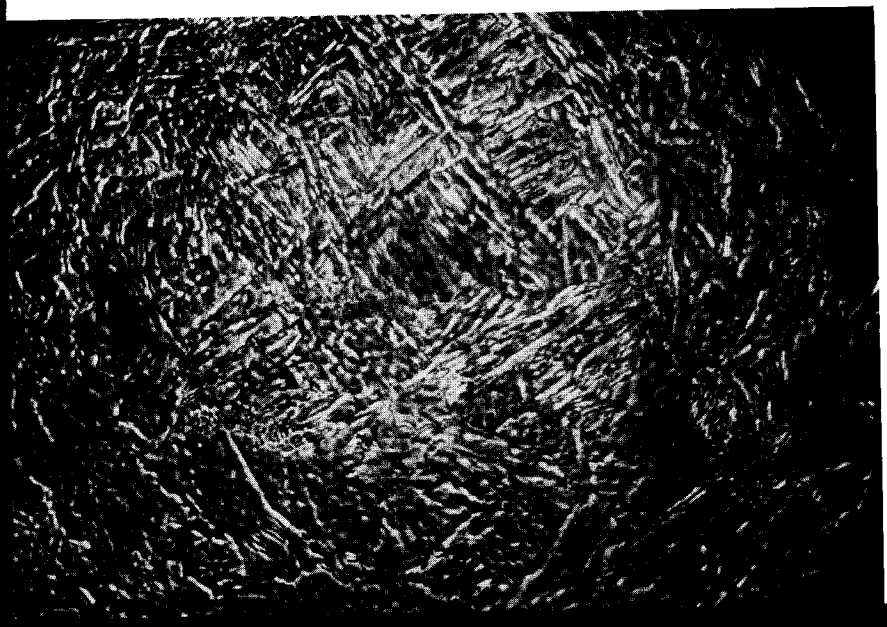

Figure 13
Same spot as in micrograph *D2* (Figure 12) with phase-contrast illumination.

D3
Magnification ×1000
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not give a clear indication of the majority of the structure. The phase-contrast technique, however, as seen in photomicrograph *C*3 (Figure 10) supplies the answer and shows more details. The structure consists of two phases: lower bainite plates with the characteristic orientation (see arrows) and a matrix of upper bainite. Here the phase-contrast illumination not only reveals more detail, but actually presents a better and more complete interpretation of the microstructure than does the conventional technique.

Figures 11 to 13 present the core structure in the tenon of a stud manufactured from AISI C 1213 steel. Micrograph *D*1 (Figure 11) shows the general area with the hardness of approximately Knoop 250, equivalent to Rockwell C 20. The center portion of this area is seen in micrographs *D*2 and *D*3 (Figures 12 and 13) with bright-field and phase-contrast illumination, respectively. Comparison of the two latter pictures reveals a characteristic property of phase-contrast technique, which is important for proper representation of the structure. Photomicrograph *D*2 (Figure 12) shows areas of free ferrite on various parts of the section. These areas are not clearly seen in the phase-contrast picture *D*3 (Figure 13) because of a substructure inside the ferrite grains. Nevertheless, the grain boundaries are definitely marked, and the substructure is different from the normal grain structure. The upper bainite plates and pearlitic matrix can more easily be identified in Figure 13, although complete understanding of the microstructure requires an examination with both conventional and phase-contrast techniques.

The phase-contrast method as yet is considered mainly as a technique in transparent specimen microscopy. There is little in the literature on the application of this method to opaque-type specimens.[2,3] Nevertheless, this technique can be used to advantage in cases where conventional microscopy does not give a complete explanation of structure and properties.

It must be emphasized that phase contrast is only a supplementary tool and should always be used in conjunction with the common bright-field illumination technique. There are cases (as shown with specimen *C*) when phase contrast in itself seems able to supply a complete answer about the microstructure. In all other specimens examined, the phase-contrast method only revealed more details in certain microconstituents or helped to clarify a confusing picture which was obtained by the conventional method. It should also be pointed out that there exists a frequent risk of misinterpretation when phase contrast is used as an independent examination method. One reason for this is that substructure appears in certain microconstituents with phase-contrast illumination (as illustrated with specimen *D*). Another more general reason is that few comparative investigations have been made between conventional and phase-contrast methods and interpretation of a structure shown on the phase-contrast microscope is often uncertain.

More frequent utilization of this method in the future will provide more material and reveal new territories in which it can be used to advantage. The field of phase-contrast microscopy is restricted; it has much promise, however, when considered as an extension or supplement to current methods of microscopic evaluation of metals.

References

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3. R. L. Seidenburg and J. R. Benford, "Phase Contrast Metallography," *Metal Progress*, **58**, 725-728 (1950).

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