NASA TECHNICAL NOTE

NASA TN D-7578

NASA TN D-7578

CASE FILE COPY

TEXTURING AND RESIDUAL STRESS IN METALS AS A RESULT OF SLIDING

by Donald R. Wheeler

Lewis Research Center

Cleveland, Ohio 44135

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • FEBRUARY 1974

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
NASA TN D-7578		
4. Title and Subtitle TEXTURING AND RESIDUAL STRESS IN METALS AS A RESULT OF SLIDING		5. Report Date February 1974
		6. Performing Organization Code
7. Author(s) Donald R. Wheeler		8. Performing Organization Report No. E-7734
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135		10. Work Unit No. 502-01
		11. Contract or Grant No.
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		13. Type of Report and Period Covered Technical Note
		14. Sponsoring Agency Code
15. Supplementary Notes		

16. Abstract

Pole figures of the texturing produced by friction in the alloys Cu - 10 at. % Sn, Cu - 10 at. % Si, 440C stainless steel, and AISI 52100 bearing steel were obtained with an energy-dispersive X-ray diffractometer. While the texturing behavior of these alloys is generally similar to that of pure metals, the 52100 steel and the Cu_5Si phase of Cu - 10 at. % Si show no texturing at the loads and speeds used in this experiment.

Photographic methods were used in an attempt to measure the uniform residual stress in the wear tracks produced on some pure metals. The stress in copper and iron was, however, below the limit of detectability - about 4.3×10^7 N/m^2 (6×10^3 psi). Line broadening under all test conditions in the case of copper and at high load and speed conditions in iron is attributed, at least in part, to the reduction of crystallite size and, perhaps, to nonuniform residual stress, as well.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Texture; Friction; Wear; Energy-dispersive X-ray diffraction; Residual stress	Unclassified - unlimited Cat.15

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price*
Unclassified	Unclassified	15	$2.75

* For sale by the National Technical Information Service, Springfield, Virginia 22151

TEXTURING AND RESIDUAL STRESS IN METALS AS A RESULT OF SLIDING

by Donald R. Wheeler

Lewis Research Center

SUMMARY

In order to determine whether the texturing produced in some alloys is similar to that produced in pure metals, sliding friction experiments with a hemispherical rider on a flat disk were performed on copper - 10 atomic percent tin, copper - 10 atomic percent silicon, AISI 52100 bearing steel, and 440C stainless steel. The wear tracks on the disks were examined by X-ray analysis with the use of an energy-dispersive diffractometer. The pole figures obtained are generally similar to those obtained for pure metals, with the texture plane being (111) in face-centered cubic structures and (110) in body-centered cubic structures. There is usually a smaller tilt towards the sliding direction, and 52100 steel and the intermetallic γ phase (Cu_5Si) of copper - 10 atomic percent silicon show no significant texture.

X-ray measurements were also made to determine the degree of residual stress produced in some pure metals by sliding. The residual uniform stress in iron and copper was below the limits of detectability of the X-ray diffraction methods used - 4.3×10^7 newtons per square meter (6×10^3 psi).

An X-ray line-broadening effect, which limited the accuracy of measurement of uniform stress, was observed in copper and at high load and speed conditions in iron. This may be due to the production of small crystallites (approx. 0.1 μm in iron) during severe wear. However, nonuniform residual stress may be a contributing factor, especially in copper.

INTRODUCTION

Whenever a metal is worked, the individual crystals tend to take on a common orientation in response to the stress. The phenomenon is called texturing, and the type and degree of texture may vary with the method of stressing the material (ref. 1). While texturing is usually associated with severe processes such as rolling, it has been observed to occur in many pure metals as a result of abrasion (ref. 2) or sliding (refs. 3 and 4). One part of this study was conducted to determine whether texturing as a result of sliding also occurs in some alloys of current or potential interest for bearing applications and whether the texturing is substantially the same as in the pure metals.

Residual stress also occurs in worked metals. It is thought that under conditions of applied stress, individual crystallites of the metal orient themselves to minimize the strain energy in the metal. When the stress is released, not all of the crystallites return to their unstrained orientations and, as a result, many crystals are under stress from the surrounding material, even after the applied stress is removed. This is known as residual stress.

By determining the difference in the lattice parameters of the worked material and the unworked material by X-ray analysis, it is possible to measure the residual strain in the metal. From this, residual stress may be calculated. The second part of this study was an attempt to use X-ray techniques to measure the residual stress produced by sliding in pure metals.

Since the two phenomena being dealt with in the two parts of this work are different, and the metal systems being studied are different, it is convenient to discuss each part of the investigation separately. Accordingly, the texturing work is discussed first, followed by the discussion of the residual stress work. A common concluding section is presented at the end of the overall report.

TEXTURING IN ALLOYS AS A RESULT OF SLIDING

The phenomenon of texturing and methods for its measurement are described in detail in reference 5 and in other literature. Here, only the experimental details unique to the present work are reported.

MATERIALS

The disk and rider specimens were made of copper - 10 atomic percent tin (Cu - 10 at. % Sn), copper - 10 atomic percent silicon (Cu - 10 at. % Si), AISI 52100 bearing steel, and 440C stainless steel. The disks were prepared by grinding successively on 240-, 300-, 400-, and 600-grit metallurgical papers, diamond polishing, and finishing with 0.3-micrometer alumina. The riders were filed to 0.475-centimeter radius and ground with 240-, 300-, 400-, and 600-grit papers and finished with 1.0-micrometer alumina.

APPARATUS

The sliding experiments were done in a common pin-and-disk apparatus described elsewhere (ref. 4). The apparatus was capable of maintaining an inert argon atmosphere around the specimens during sliding.

An X-ray diffractometer was required for these measurements. The diffractometer used was constructed for the purpose and is unusual in principle. Its main features will be described.

The Bragg condition is that a crystal plane with spacing d will reflect X-rays of wavelength λ at angle θ, if

$$\lambda = 2d \sin \theta$$

In the operation of a typical diffractometer, a beam of X-rays of one wavelength is directed at a sample and θ is varied to observe reflections corresponding to the various d spacings of the crystals. With the use of the Bragg equation, the d spacings are calculated from the known wavelength λ and the observed diffraction angle θ.

The recent availability of lithium-drifted silicon, Si(Li), X-ray detectors of very good energy resolution has made it possible to approach the problem differently. The sample is illuminated at a fixed angle θ by a beam of essentially "white" radiation. Each properly positioned plane in the sample then reflects radiation of the appropriate wavelength λ. Other wavelengths are reflected weakly or not at all. The detector, together with a multichannel analyzer, displays the energy spectrum of the scattered radiation. Each peak in the spectrum is due to diffraction by a plane of spacing d. If the Bragg condition is rewritten in terms of energy,

$$\frac{hc}{E} = 2d \sin \theta$$

where h is Planck's constant, and c the speed of light, the spacing d can be calculated from the known angle θ and the observed energy of diffraction E. This technique, known as energy-dispersive diffractometry, was used for this study.

The advantages of the technique and some important considerations for its use have been described elsewhere (refs. 6 and 7). In this study, the primary advantage was ability to see all the diffraction peaks at one time. Thus, texturing observed on one plane could be confirmed by observations on crystallographically related planes, and in the case of the copper-silicon alloy, the texture behavior of both coexisting phases could be measured simultaneously.

In our apparatus, the X-rays from a chromium target tube were collimated to a beam approximately 1 millimeter in diameter at the sample. The beam was incident on the sample at a Bragg angle of 15°. A second collimator in front of the Si(Li) detector served to define the angle of reflection at 15°. The sample disks were mounted in a special jig on a standard crystal-orienter goniometer. The goniometer axes passed through the wear track on the disk so that the wear track could be tilted in two directions to obtain pole figure data. The jig incorporated a small electric motor which continually rotated the sample about its own axis. Thus, the measured intensities are effectively averaged over all positions on the wear track.

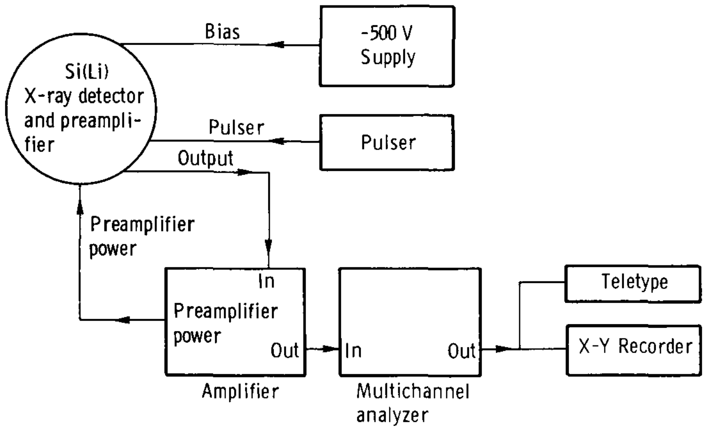


Figure 1. - Block diagram of energy-dispersive diffractometer electronics.

Figure 1 is a block diagram of the electronics required to detect and record the diffraction pattern. For each goniometer setting, the entire diffraction pattern in the range from 10 to 30 keV was recorded on the teletype. For the samples used here with $\theta = 15^{\circ}$, this range included all the diffraction lines of interest. X-ray fluorescence lines from the samples were observed below 10 keV and served to verify, qualitatively, the compositions of the samples.

Figure 2 shows a typical diffraction pattern obtained from a pure copper sample. This sample was subjected to sliding against a copper rider at 5.18 centimeters per second for 1000 passes with a 300-gram-weight load. A pole figure showed all the features previously obtained with a conventional diffractometer (ref. 4).

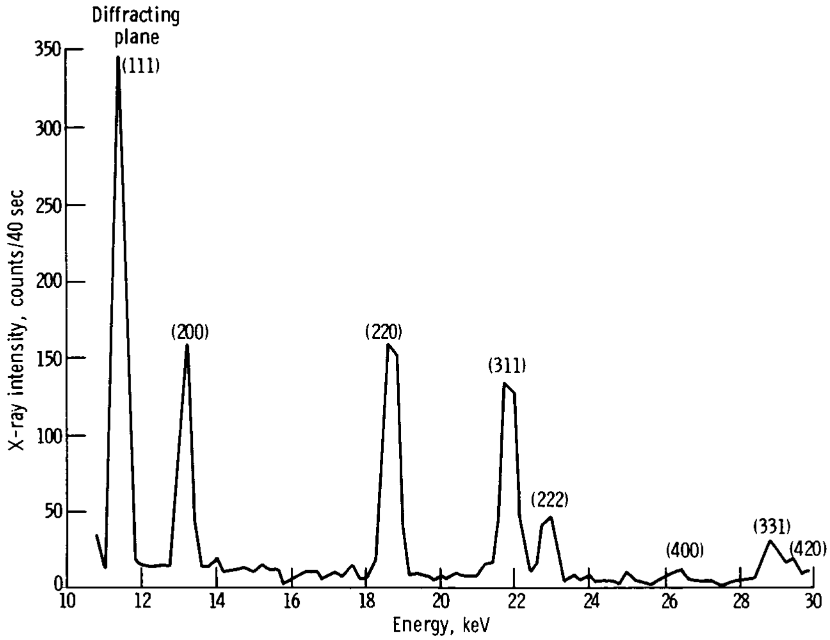


Figure 2. - Typical energy-dispersive diffraction pattern from copper wear track (10 to 30 keV region).

PROCEDURE

Sliding experiments on all samples were performed in an argon atmosphere. A pure copper rider was used with the two copper-based alloys. With the iron-based alloys, the riders were the same composition as the disks. The sliding speed was 5.18 centimeters per second in all cases, and the load was 500 grams weight. Sliding was continued for 1200 passes. Because the X-ray beam spot on the disks was several millimeters long at a projection angle of 15°, a series of adjacent concentric wear tracks was produced on each disk. In this way, it was assured that only the worn area was being illuminated by the beam.

The data obtained were used to plot pole figures of the usual slip planes for each material. Because of counting statistics and some instrumental fluctuations in peak intensity, texture peaks with heights less than 10 percent of the background height are not significant. Such peaks are reported as "no significant texturing" and pole figures are not shown. Such negative results are valid only for the relatively mild conditions of this experiment. Texturing may occur in these metals at high enough loads.

RESULTS

AISI 52100 Bearing Steel

No significant texture was observed. This sample showed noticeably less wear than any of the others. Again, increasing the load may have brought about the development of a surface texture.

440C Stainless Steel

Figure 3 shows the pole figure for the (110) plane in this sample. The lateral spread of the contours is due to ridges in the wear track parallel to the sliding direction. It has been observed in other studies (refs. 3 and 4). The lobed effect is similar to, but more pronounced than, that observed in pure iron (ref. 4).

Copper - 10-Atomic-Percent-Tin Alloy

Figure 4 is the pole figure for this alloy. A pronounced (111) texture is evident. The elongation in the sliding direction may be a surface roughness artifact, or it may be an aspect of the tilt in tne sliding direction, which has been observed in pure metals (refs. 3 and 4).

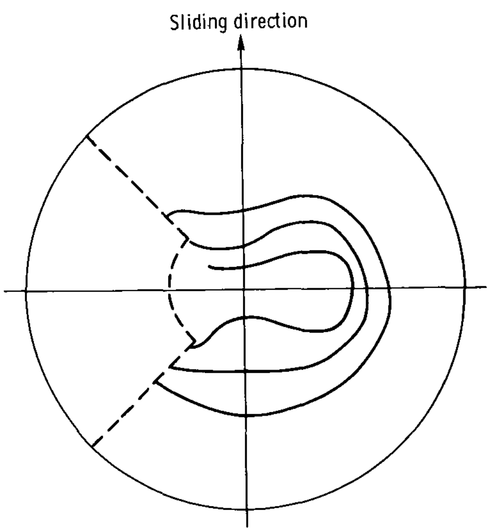


Figure 3. - Pole figure of (110) diffracting plane of 440C stainless steel after wear. Dashed line represents boundary of observed region of projection.

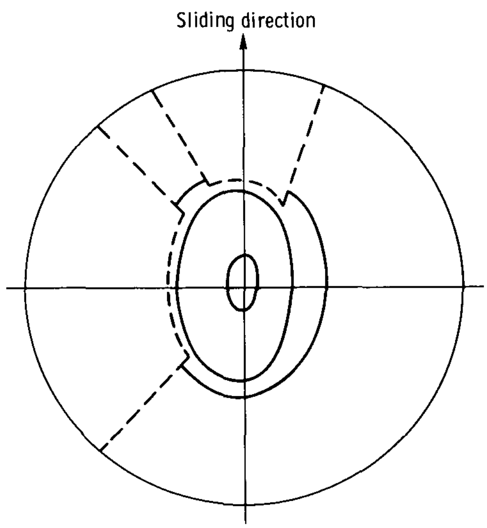


Figure 4. - Pole figure of (111) diffracting plane of copper - 10-atomic-percent-tin alloy after wear.

Copper - 10-Atomic-Percent-Silicon Alloy

This alloy forms a mixture of pure copper and the intermetallic γ phase, Cu_5Si (ref. 8). The observed diffraction pattern was indexed on this basis (ref. 9). Separate pole figures were prepared for Cu and Cu_5Si. The Cu_5Si shows no significant texture.

The pole figure for copper in this mixture is shown in figure 5. The texture peak is

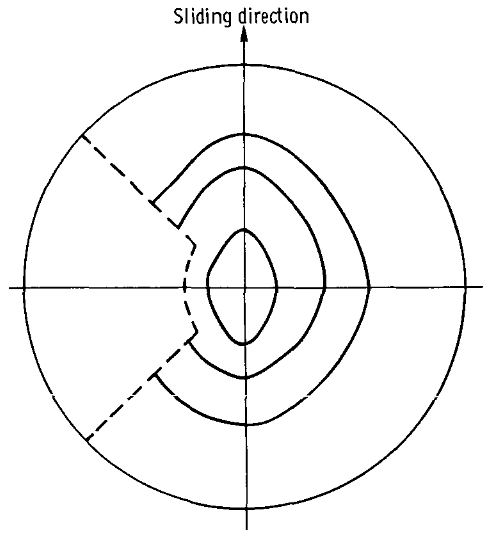


Figure 5. - Pole figure of (111) diffracting plane of copper after wear of two-phase (Cu-Cu_5Si) copper - 10-atomic-percent-silicon alloy.

broader than that observed in the wear of pure copper and is not shifted in the sliding direction. The true behavior of the Cu (111) is obscured, however, by the Cu_5Si (300) peak, which is superimposed on it. The absence of lateral spread may be due to the lack of severe surface damage in this sample.

RESIDUAL STRESS IN PURE METALS AS A RESULT OF SLIDING

X-ray analytical methods for the measurement of stress and residual stress in crystalline materials are in common use (ref. 5). The apparatus and procedures used herein conform to the usual practice. The stress in a particular direction in the surface is determined by measuring the spacing $d_\perp$ of planes nearly perpendicular to the surface and the spacing $d\varphi$ of the same planes in crystals whose normals are turned by some angle φ about an axis parallel to the surface and perpendicular to the direction of the component of stress desired. The component of stress is then

$$\sigma = \frac{d_{\perp} - d_{\varphi}}{d_{\perp}} \frac{E}{\nu} \frac{1}{\sin^2 \varphi}$$

where E is Young's modulus, and ν is Poisson's ratio for the material.

In practice, the X-ray beam is first directed normal to the surface, and a diffraction ring in the back reflection region ($\theta \geq 75^{\circ}$) is measured. The spacing of the plane producing this ring is taken to be $d_{\perp}$, although the plane is actually 5° to 15° from true perpendicular. The sample is then tilted by an amount $\varphi = 30^{\circ}$ to 45° about the appropriate axis, and the same ring is remeasured to get d_{φ}.

The uniform stress, or macrostress, measured by line shift must be distinguished from nonuniform stress, or microstress. Usually, both are present in plastically deformed materials. In nonuniform stress, the grains are deformed in various directions. The result is a range of lattice spacings, some greater and some less than normal. The X-ray lines produced in this case are broadened, and a measure of this broadening can be used to calculate the maximum tensile (or compressive) stress present:

$$\sigma_{max} = \frac{Eb}{4 \tan \theta}$$

where b is the broadening expressed as twice the range of Bragg angle, in radians, covered by the line ($b = 2\Delta\theta$).

Finally, it must be kept in mind that line broadening is also produced by small crystallite size. The size t of crystals producing a line of breadth b is, approximately,

$$t = \frac{0.9 \lambda}{b \cos \theta}$$

MATERIALS

Rider and disk combinations of 99.998-percent-pure copper and of 99.95-percent-pure iron were used. The specimens were prepared in the same manner as for the texturing studies. In addition, they were etched with 5 percent nitric acid in ethyl alcohol.

APPARATUS

The friction-and-wear device was the same pin-and-disk apparatus that was used for the texturing experiments. In this second part of the investigation, the disk and rider were always of the same material. Deadweight loading of the rider against the disk was variable up to 500 grams weight, and the speed was variable from 5.18 centimeters per second.

A choice of chromium, iron, cobalt, copper, and molybdenum target tubes was available. These represent most of the X-ray wavelengths that are useful for diffraction work.

A back-reflection Laue camera with a Polaroid XR-7 cassette was used with Polaroid 3000-speed film to detect the X-rays. Lead masks permitted exposure of opposite quadrants of the film separately. A special jig held the specimen disks and allowed them to be positioned perpendicular to or at 45° to the X-ray beam. The axis of rotation was perpendicular to the beam and tangent to the wear track. The beam could be directed onto the wear track or onto the unworn disk surface. In order to produce smoother diffraction rings, provision was made to oscillate the disk through an angle of about 10° around its own axis.

PROCEDURE

Specimens were subjected to sliding friction under the desired conditions in the friction-and-wear apparatus. The disks were then mounted in the X-ray camera with their surfaces perpendicular to the beam. Opposite quadrants of the film were covered with the masks, so that only half the film was exposed. An exposure was then made - 40 minutes usually being sufficient. Next, the sample was turned 45°, and the other two film quadrants were exposed. A difference in the diameters of the two partial diffraction rings produced in this way indicates a difference between $d_{\perp}$ and d_{φ} and thus a component of stress in the direction of wear.

If the axis about which the sample was rotated lay off the surface of the sample, the rotation would change the distance between the sample and the film. This would produce a change in the ring diameters and would lead to a spurious conclusion. To avoid this, a second set of exposures was made with the beam directed off the wear track onto the polished and etched, and presumably unstressed, surface. Any difference in the ring diameters observed in this case could be used as an instrument correction in interpreting the first set of exposures.

RESULTS

Iron

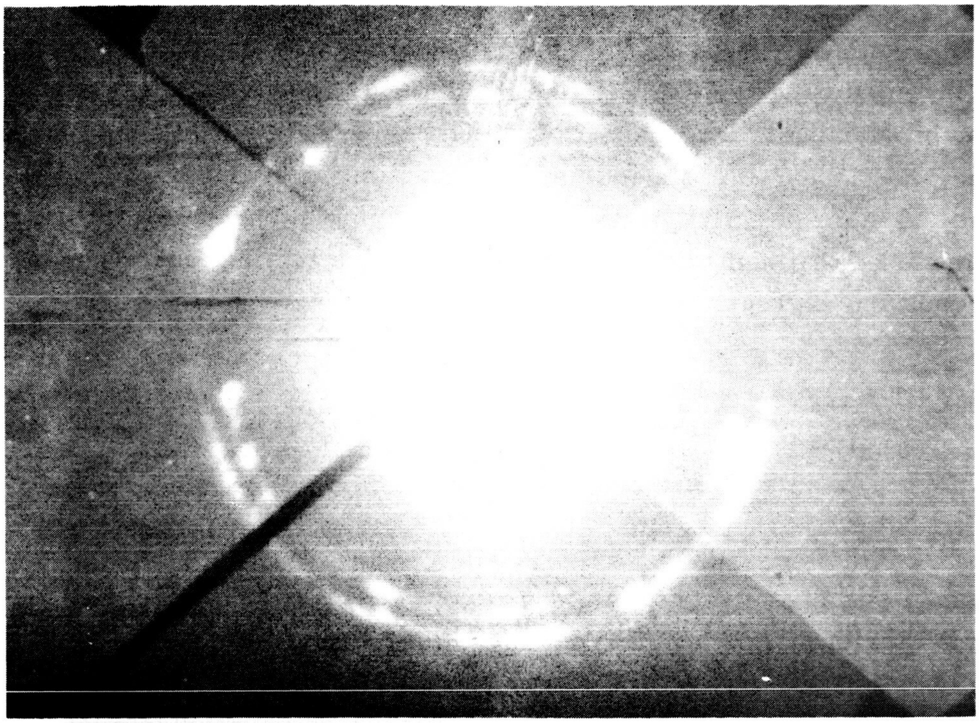
A typical diffraction pattern for iron is shown in figure 6(a). Within the limits of error of measurement (±0.5 mm), there is no difference in the diameters of the diffraction rings between the normal and the 45^{o} segments. With 0.5 millimeter used as the maximum difference, the upper limit to the residual uniform stress in the wear direction is calculated to be 4.32×10^{7} newtons per square meter (6.16×10^{3} psi). A Young's modulus of 9.25×10^{10} newtons per square meter (13×10^{6} psi) and Poisson's ratio of 0.27 have been used for iron. The iron continues to give patterns identical to figure 6(a) at all loads through 500 grams at 5.18 centimeters per second and at 24.8 centimeters per second with loads below 500 grams. However, at 24.8 centimeters per second and 500 grams. the pattern shown in figure 6(b) was obtained. The diffuse nature of the rings increases, somewhat, the uncertainty of measurement. The upper limit for the uniform residual stress in this case is 8.7×10^{7} newtons per square meter (12.3×10^{3} psi).

The diffuse appearance of the rings in figure 6(b) can be due to either nonuniform stress or small crystallite size, or both. Nonuniform stress is a common feature of cold-worked metals. The line width in this case is clearly greater than the separation of the $K_{\alpha 1}$ and $K_{\alpha 2}$ lines. With this separation used as a lower limit, a maximum nonuniform residual stress of at least 3.92×10^{7} newtons per square meter (5.5×10^{3} psi) is calculated. The rather smooth nature of the lines in figure 6(b), compared to those in figure 6(a), indicates that recrystallization and the associated reduced crystallite size contributes significantly to the observed line broadening. All of the observed broadening could be due to particle sizes of 0.1 micrometer. Such small sizes have been observed in cold-worked metals (ref. 10). In fact, a combination of nonuniform stress and size effects probably produce the broadening observed, but these data do not justify further speculation.

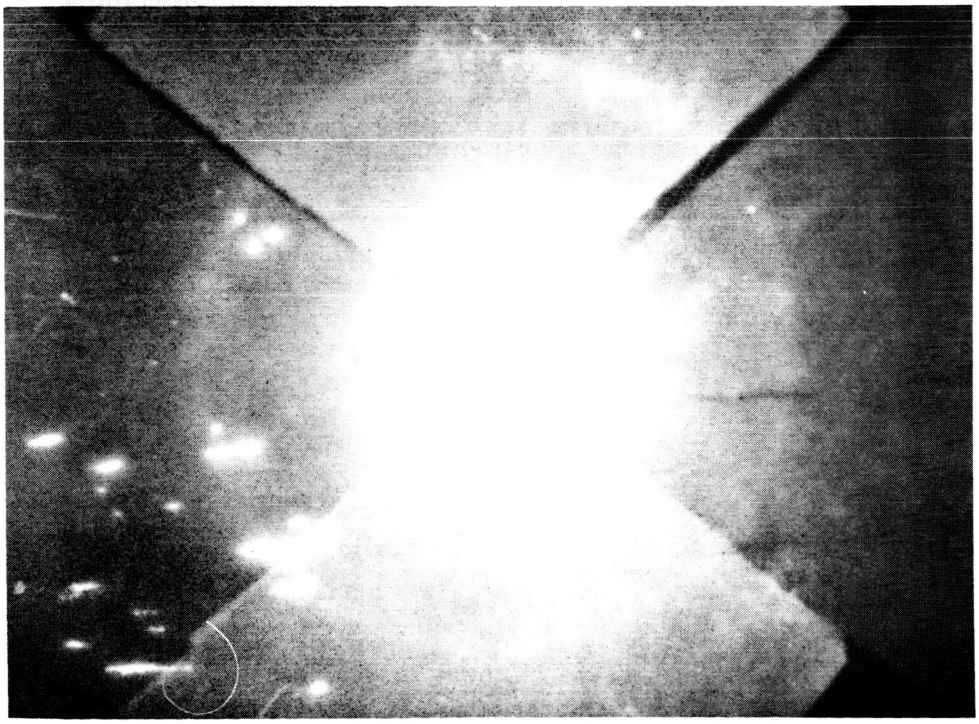
Copper

The (400) cobalt K_{α} diffraction ring of the etched copper surface was sharp and easily measured. However, the pattern obtained from the wear track at the lowest possible speed (5.18 cm/sec) and load (approximately 50 g) after 100 passes was so diffuse as to be unmeasureable. These conditions were the mildest that would produce a wear track at least the size of the analyzing X-ray beam. More extreme wear conditions broadened the line to the point that it was lost in the background.

It is not possible to draw quantitative conclusions from these copper results. Some



(a) Etched surface.



(b) Wear track.

Figure 6. - Iron (310) diffraction line from cobalt K_α radiation. Angle between film plane and specimen surface for horizontal quadrants, 0°; for vertical quadrants, 45°.

qualitative conclusions, however, can be drawn. Since particle sizes of atomic dimensions would be required to produce the most severe line broadening observed, this possibility may be ruled out. We are left, then, with large nonuniform strains as the cause. Since the line broadening is inversely proportional to Young's modulus and proportional to the stress, and since Young's modulus for copper is roughly half that for iron, we see that the stress is more effective in producing broadening in copper than in iron. Still, it must be concluded that higher nonuniform stresses are retained in copper even under more mild conditions of wear than in iron.

SUMMARY OF RESULTS

TEXTURING

An energy-dispersive X-ray diffractometer was used to obtain pole figures of four alloy samples after they had been subjected to sliding in a pin-and-disk apparatus at a speed of 5.18 centimeters per second, under a load of 500 grams weight, for 1200 passes. The following observations were made from these pole figures:

1. Texturing is caused by the sliding process in copper - 10 atomic percent tin (Cu - 10 at. % Sn) and in 440C stainless steel. In the face-centered cubic alloy (Cu - 10 at. % Sn), the texture plane is (111); and in the body-centered cubic alloy (440C stainless steel), it is (110).
2. The unusually hard, AISI 52100 bearing steel showed no texture at the loads used in this study.
3. The copper in the two-phase (Cu-Cu_5Si) copper - 10-atomic-percent-silicon alloy shows a texture much like pure copper. The intermetallic γ phase (Cu_5Si) does not texture.

RESIDUAL STRESS

Photographic techniques were used to study the X-ray diffraction lines produced by some pure metal samples that had been subjected to sliding. Both the shift and the broadening of the lines due to various degrees of wear were observed. The following are the principal results of this study:

1. In iron, a line broadening is evident under severe wear conditions (load, 500 g wt.; sliding speed, 24.8 cm/sec). This line broadening can be attributed to small particle size (0.1 μm) or to nonuniform residual stress of 3.92×10^7 newtons per square meter (5.5×10^3 psi).

2. In copper, the line broadening is large under the most mild wear conditions. This line broadening is probably due to nonuniform stresses, perhaps an order of magnitude greater than those in iron.

3. Line broadening is generally pronounced. It would be the most sensitive way of studying the residual stress due to wear in metals. Quantitative analysis of line profiles would permit the separate evaluation of particle size distribution and residual stress.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, November 13, 1973,
502-01.

REFERENCES

1. Barrett, Charles S.: Structure of Metals. McGraw-Hill Book Co., Inc., 1943.
2. Scott, V. D.; and Wilman, H.: Surface Re-Orientation Caused on Metals by Abrasion - Its Nature, Origin and Relation to Friction and Wear. Proc. Roy. Soc. (London), Ser. A, vol. 247, no. 1250, Sept. 30, 1958, pp. 353-368.
3. Huppman, W. J.; and Clegg, M. A.: The Tribological Behavior of Polycrystalline Cobalt as Related to Crystallographic Texture and Structure. Paper 72LC-5B-1, ASLE, Oct. 1972.
4. Wheeler, Donald R.; and Buckley, Donald H.: Texturing in Metals as a Result of Sliding. NASA TN D-7136, 1973.
5. Klug, Harold P.; and Alexander, Leroy E.: X-ray Diffraction Procedures for Polycrystalline and Amorphous Materials. John Wiley & Sons, Inc., 1954.
6. Giessen, Bill C.; and Gordon, Glen E.: X-ray Diffraction: New High-Speed Technique Based on X-ray Spectrography. Science, vol. 159, no. 3818, Mar. 1, 1968, pp. 973-975.
7. Sparks, Cullie J., Jr.; and Gedcke, Dale A.: Rapid Recording of Powder Diffraction Patterns with Lithium-Drifted Silicon X-ray Energy Analysis Systems. Tungsten and Copper Targets and Error Analysis. Advances in X-ray Analysis. Vol. 15. Plenum Publ. Corp., 1972, pp. 240-253.
8. Hansen, Max; and Anderko, Kurt: Constitution of Binary Alloys. Second ed., McGraw-Hill Book Co., Inc., 1958, p. 630.
9. Joint Committee on Powder Diffraction Standards: Powder Diffraction File Card 4-0841. ASTM, 1960.

10. Warren, B. E.: X-Ray Studies of Deformed Metals. Progress in Metal Physics. Vol. 8. Bruce Chalmers and R. King, eds., Pergamon Press, 1959, pp. 147-202.

11. Cullity, Bernard D.: Elements of X-ray Diffraction. Addison-Wesley Publ. Co., 1956.

NASA-Langley, 1974 E-7734