

Communications

Glasslike Behavior in a Nanostructured Fe/Cu Alloy

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In this article, we report the observation of shear bands and perfectly plastic behavior, typical of metallic glasses, in an Fe-Cu alloy with grain sizes in the 100 nm range. A strengthening effect with decreasing grain size was also observed, and the shear bands occurred on planes oriented at about 40 deg from the tensile axis instead of the maximum shear plane of 45 deg, as has been observed in amorphous polymers.

Fe-Cu alloys containing 10 wt pct copper were prepared by ball milling of iron (99.9 pct) and copper (99.5 pct) powders in a Svegari attritor system. 440C hardened stainless steel media (6.75 mm diameter) were used at a weight ratio of 10:1. To limit contamination, the system was contained in an argon glove box with controlled moisture (0.03 ppm) and oxygen (<2.5 ppm) levels. Milling was conducted at 550 rpm for 24 hours, with the coolant temperature maintained below 0 °C. X-ray diffraction (XRD) analysis after milling revealed the presence of a single-phase, bcc iron. Changes in lattice parameter of the milled powder indicated that copper was present in extended solid solution. As-milled grain size was determined by line-broadening techniques using the Scherrer equation^[1] and the Warren-Averbach method,^[2] which were found to agree for the (110) peak at a grain size of 18 nm. Pure Fe powders were also prepared under identical conditions for comparison. As-milled grain size of the pure Fe was 22 nm.

The powders were consolidated by hot isostatic pressing (HIP) at 170 GPa for 30 minutes at temperature and pressure. Two temperatures, 600 °C and 700 °C, were investigated to compare consolidation behavior, coarsening kinetics, and mechanical properties. The densities of the compacts were determined with the Archimedes technique to be greater than 99 pct of theoretical density for both samples. The pure Fe powders processed under identical conditions resulted in near 100 pct density at 700 °C but only 98 pct at 600 °C. The XRD analysis and transmission electron microscopy (TEM) selected area diffraction revealed only two phases: bcc iron and fcc copper. Lattice parameter values indicated that the copper came completely out of solution during consolidation.

As-HIP grain sizes and microstructures were studied by X-ray and TEM techniques. Transmission electron microscopy foils were prepared by ion milling, due to difficulties associated with different electropolishing rates of Fe and Cu. Analysis of multiple TEM foils, including the meas-



Fig. 1—Representative TEM microstructure of the Fe-10 pct Cu (700 °C HIP) specimen. Likely copper grains, based on numerous dark-field images taken in similar foils, are indicated by arrows.

Table I. Mechanical Properties

Alloy, HIP Temperature	σ_y , MPa	Vickers Hardness, GPa
Fe, 700 °C	495, 525, 540	2.2
Fe-10 pct Cu, 700 °C	1140, 1140, 1150	3.3
Fe-10 pct Cu, 600 °C	1700, 1710, 1730, 1740	4.6

urement of several hundred grain diameters, indicated that both the Scherrer equation and the Warren-Averbach approach severely underestimated the iron grain size for the consolidated hot isostatically pressed samples. Whereas X-ray techniques indicated average copper and iron grain sizes in the 50 to 100 nm range depending on the alloy and processing temperature, TEM techniques found much larger grain sizes. The TEM analysis determined that the two-phase mixture obtained by HIP at 700 °C consisted of Fe grains with a broad distribution in the 50 to 600 nm range, along with Cu grains in the 10 to 150 nm range, usually in triple junctions and Fe/Fe grain boundaries. Copper grains were identified by dark-field imaging using the (220) copper reflection, which can be easily isolated from iron reflections. The median grain size, including both Cu and Fe, was determined by TEM to be 130 nm, with 90 pct of the grains being less than 350 nm. After HIP at 600 °C, the median grain size was 100 nm, with 90 pct of the grains being less than 250 nm. A typical micrograph is shown in Figure 1. Based on our work with pearlitic steels (electropolished and ion milled), we have concluded that most of the mottling contrast seen in the micrographs is due to ion milling damage. The pure iron (700 °C HIP specimen) exhibited much larger grain sizes, in the tens of micrometers.

Rectangular compression specimens, approximately $5 \times 5 \times 10$ mm, were prepared from the compacts. At least three compression tests were conducted for each condition, and Vickers hardness values were also measured. Mechanical test results are presented in Table I, and representative stress-strain curves are shown in Figure 2. Pure Fe (700 °C HIP)

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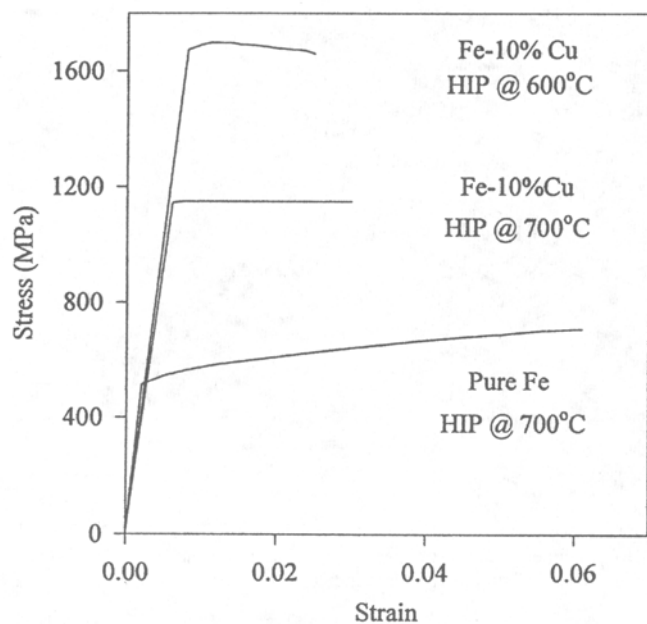


Fig. 2—Stress-strain curves obtained in compression.

demonstrated traditional work-hardening behavior. Pure Fe (600 °C) was not tested since it was less than 99 pct of theoretical density. The 10 pct Cu (700 °C HIP) sample behaved in a perfectly plastic manner; that is, the behavior was elastic, followed by a distinct yield point and plasticity with no strain hardening. The 10 pct Cu (600 °C HIP) sample behaved similarly, except the strength was 50 pct higher, and the material softened slightly during plasticity, as evidenced by the decreasing stress-strain curve. The strength values, especially for the alloys, show very little scatter. The reported hardness values are averages of ten impressions.

Plastic deformation occurred by shear banding, as evidenced by slip traces on the specimen surfaces and discrete shear offsets. Figure 3 shows a typical specimen. There are two sets of major, planar shear bands, as well as some finer, more “wavy” bands. A shear offset due to the major band is clearly present. The average angle between the shear band plane normal and the stress axis was 41 deg, which is not the plane of maximum shear. The 10 pct Cu (600 °C HIP) specimens deformed similarly, except the shear band width was finer and the number of bands was higher. These deformation bands are not Lüders bands but shear bands. This is evident because there was no load drop, the banding did not occur in the pure iron samples, and the bands did not grow in width across the specimen gage length as Lüders bands do in low-carbon steels. Further, the shear offsets observed for the major bands (Figure 3) were macroscopically large, whereas the local strains observed due to Lüders bands are typically much smaller.

The 10 pct Cu (700 °C HIP) samples did not fracture but started to buckle after about 5 pct plastic strain. The 10 pct Cu (600 °C HIP) specimens failed by shear fracture along a major shear band after about 2 to 3 pct plastic strain.

The behavior observed in these nanocrystalline iron-copper alloys is quite different from traditional metals. Shear bands are observed in polycrystalline metals, but only after large amounts of generalized plasticity and/or at shock-loading strain rates.^[3,4,5] In this study, shear bands occurred as the first mechanism of plastic deformation. Although not

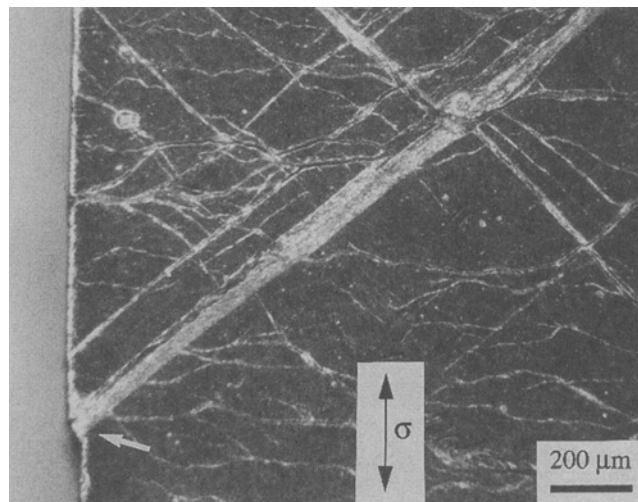


Fig. 3—Optical micrograph of an Fe-10 pct Cu (700 °C HIP) specimen, showing intersecting shear bands and shear offset on specimen surface (arrow). Note that shear bands do not intersect at a 90 deg angle, and shear band planes are not inclined at 45 deg to the stress axis.

typical for metals, shear banding at the yield point and perfectly plastic behavior (or softening) are commonly observed in metallic glasses^[6] and amorphous polymers.^[7,8] Further, amorphous polymers which deform by shear banding typically exhibit shear bands on planes inclined to the stress axis at angles around 38 deg, in the same sense of inclination as observed here.^[7,8]

The similarities between the amorphous materials and the alloys discussed here are surprising; the grain sizes of the specimens, while small relative to traditional steels, are not in the range where one would expect glasslike behavior. In the 5 nm grain size range, it might be expected that the disordered character of the boundaries could lead to glasslike behavior,^[9] because the volume fraction of “boundary” atoms is significant. Indeed, Niemann *et al.*^[10] have observed diffuse “shear bands,” or localized deformation, on the surfaces of nanocrystalline metal specimens in the 10 nm grain size regime. At 100 nm grain size, however, the fraction of boundary atoms is very small. If the inherent nature of the material is not strongly glasslike, then it is possible that the deformation instability resulted in a transition in material structure. Ovid’ko^[11] discussed the theory of amorphitization during plastic deformation and proposed the occurrence of amorphitization during shear band instabilities, especially at high rates. On the other hand, the current study showed that the strength of the alloy increased as the grain size decreased, an observation which is consistent with dislocation-based plasticity. Further, if amorphitization were occurring during deformation, it would be reasonable to assume that the amorphitization process would be easier at smaller grain sizes, which should result in grain size softening instead of the hardening which was observed. Another intriguing point is that the grain size is approximately the same as the subgrain size which is typically obtained in metals after significant plasticity, at the point of shear banding. Transmission electron microscopy work is in progress to study the microstructure inside the shear bands and to determine the mechanisms of plastic flow.

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Grain Structure and Quench-Rate Effects on Strength and Toughness of AA7050 Al-Zn-Mg-Cu-Zr Alloy Plate

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The fracture toughness of heat-treatable Al-Zn-Mg-Cu aluminum alloys is influenced by a number of microstructural features, which are controlled by chemistry and processing. The effects of constituent particles (insoluble and soluble) and hardening precipitates are well known and understood.^[1,2,3] Also well accepted are the independent effects of quench rate and recrystallization, whereby slack quenches reduce toughness,^[4] as do recrystallized structures.^[3,5] Less is understood, however, about possible interactions between these factors. It has been claimed that the rate of loss in toughness with decreasing quench rate is higher for recrystallized products because of precipitation on high-angle boundaries.^[6] Although this contention is partially supported by loss-of-ductility data,^[7,8] the prior results were obtained on Cr/Zr-free alloys with essentially equiaxed grain structures. And, as Unwin and Smith noted,^[9] tensile elongation in such materials does not necessarily correlate with fracture toughness.

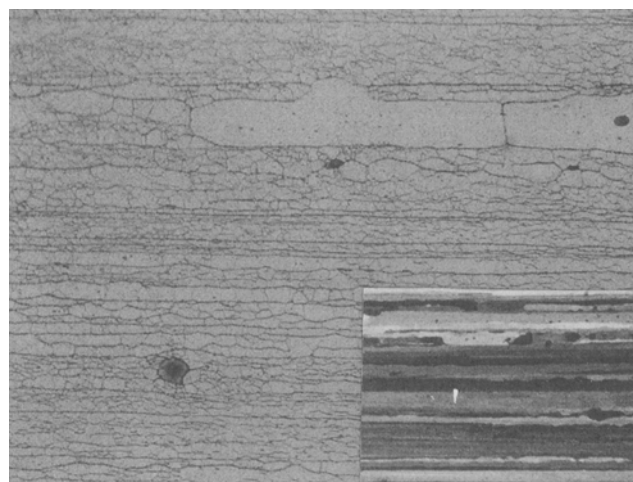
To examine the inter-relationships between quench rate and recrystallization as they affect both strength and fracture toughness, a systematic study was conducted in which AA7050 plates with various grain structures were solution

heat-treated using a number of quench rates. The starting material was a 150-mm-thick F temper plate (composition shown in Table I), which was hot-rolled to 14-mm-thick plates. By varying the final (exit) rolling temperature from 245 °C to 385 °C, levels of recrystallization ranging from 15 to 80 pct at the center of the plates were achieved after solution heat treating at 475 °C for 1 hour. Recrystallization was estimated visually after a 5 minute etch in 10 pct H₃PO₄ at 50 °C. The photomicrographs in Figure 1 show that the unrecrystallized condition is characterized by a

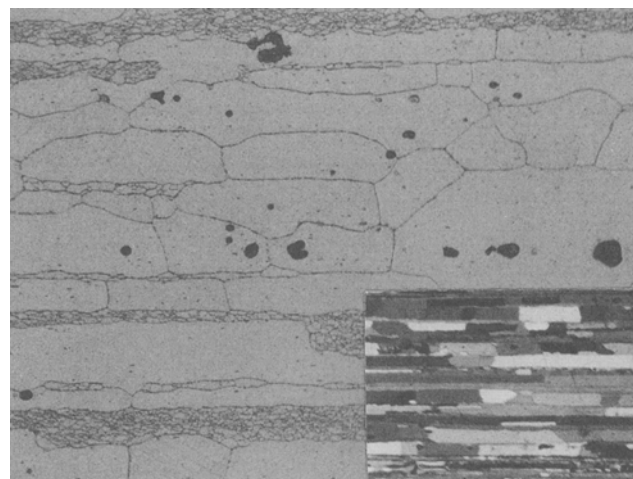
Table I. Chemical Composition of 7050 Plate

Weight Percent*						
Si**	Fe	Cu	Mg	Zn	Ti	Zr
0.04	0.07	2.25	2.11	6.43	0.04	0.10

*By inductively coupled plasma spectroscopy, except silicon (others < 0.01 pct each).
**Quantometer estimate.



20 μm
(a)



(b)

Fig. 1—Microstructures of 7050 plates: (a) 15 pct recrystallized and (b) 80 pct recrystallized. High-angle boundaries in (a) actually define relatively large elongated grains as revealed by the Barker's etch insert (1/5 magnification of the 10 pct H₃PO₄ etch photomicrographs).

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