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U. de Andrade, M. Meyers, A. Chokshi, K. Vecchio. Dynamic recrystallization and grain size effects in shock hardened copper. Journal de Physique IV Proceedings, 1994, 04 (C8), pp.C8-361-C8-366. 10.1051/jp4:1994855 . jpa-00253415

HAL Id: jpa-00253415

<https://hal.science/jpa-00253415>

Submitted on 1 Jan 1994

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Dynamic recrystallization and grain size effects in shock hardened copper

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Résumé: Des échantillons de cuivre ont été soumis à un chargement par ondes de choc de 50 GPa suivi par une haute déformation $\gamma \sim 2-5$ à une vitesse de déformation élevée ($\sim 10^4 \text{ s}^{-1}$). La structure recrystallisée consistait de grains fins et equiaxes (0.1-0.3 μm). Des essais à basse vitesse de déformation sur ces mêmes échantillons soumis au choc et préchauffés ont produit une structure recrystallisée avec une taille de grains de 2-3 μm et une densité élevée de macles de recuit. Ces différences indiquent que les mécanismes de recristallisation dynamique opérant à basses et hautes vitesses de déformation sont différents. L'effet de la taille de grain initial a été aussi étudié: des tailles de grain de 10, 5, 25, 117, et 315 μm , ont été produites. Le chargement par choc a créé une haute densité de dislocations dans les échantillons à basses tailles de grains et une densité élevée de macles de déformation dans les tailles élevées. Ces différences ont un effet prononcé sur la réponse mécanique subséquente et affectent la tendance à la localisation, l'écroutissage, et la contrainte d'écoulement du matériau.

Abstract: Copper pre-shocked to 50 GPa underwent dynamic recrystallization, when subsequently plastically deformed to high strains ($\gamma \sim 2-5$) at high strain rates. The recrystallized structure generated at high strain rates consisted of small, equiaxed grains (0.1-0.3 μm). Low-strain-rate experiments on pre-shocked (and pre-heated specimens) revealed a recrystallized grain size of $\sim 2-3 \mu\text{m}$, with profuse annealing twins; this is suggestive of different mechanisms operating at low and high strain rates. The effect of grain size (9.5, 25, 117, 315 μm) on high-strain, high-strain-rate deformation of copper was also investigated. Pre-shocking of the specimens generated a high dislocation density and deformation twins. Twinning was highly dependent on grain size, being profuse for 177 and 315 μm , and virtually absent for 9.5 μm specimens. This was observed to have a profound effect on the subsequent mechanical response of the larger grain-sized specimens, which underwent considerably larger shock-wave hardening than the smaller grain-sized ones.

1. INTRODUCTION

At the 1992 DYMAT, Meyers et al. [1] reported on the dynamic recrystallization undergone by pre-shocked copper (50 GPa) subjected to high strains ($\gamma \sim 2-5$) and high strain rates ($\sim 10^4 \text{ s}^{-1}$). These findings were consistent with earlier suggestions by Chokshi and Meyers [2] that dynamic recrystallization could have a profound effect on the stability of jet formation in shaped charges. There was early evidence that shaped charges underwent dynamic recrystallization [3] and recent work by Murr and coworkers [4,5] has supported this. The work reported herein represents a continuation of earlier investigations aimed at:

- a- identifying the mechanism of dynamic recrystallization
- b- determining the effect of grain size on the phenomena above.

Extended versions of this work have been submitted elsewhere [6,7]. For experimental details, the reader is referred to Andrade [8].

2. EXPERIMENTAL TECHNIQUES

The copper specimens (99.99%) were received as plates (19 and 51 mm thick) and a cylindrical bar (109 mm height). Different grain sizes were produced by rolling the plate down to 7mm, and by upsetting a cylinder to a 10.9 mm height. Subsequently, annealing treatments yielded the desired range of grain sizes: 673 K for 240 s $\rightarrow$ 9.5 μm ; 773 K for 1200 s $\rightarrow$ 25 μm ; 873 K for 6000 s $\rightarrow$ 117 μm ; 1073 K for 6000 s $\rightarrow$ 315 μm . Shock loading was accomplished in a fixture similar to the one described previously [1]. The impact of a 4.7 mm stainless steel plate on the system containing the specimens, at velocities of 2.2, 2.028, and 2.0 km/s (3 systems were used) yielded pressures of 4.9-5.0 GPa and pulse duration of approximately 2 μs . The velocities were determined by the pin method [8]. Special care was taken to minimize the residual strain in the specimens. Following Mogilevsky and Teplyakova's [9] suggestions, a taper was used for montage of the specimens; additionally, a steel confinement plate was used to decrease, as much as possible, the residual lateral strains in the specimens. One of the experiments was executed on a system pre-cooled to 277 K in an alcohol-dry ice bath; the specimens were recovered in a water drum. The total effective transient strain undergone by the specimens during shock loading was calculated from the equation

$$\epsilon = \frac{4}{3} \ln \frac{V}{V_0}$$

where V and V_0 are the initial and shock compressed volumes, respectively. The 50 GPa pressure yielded a total effective strain of 0.278. This corresponds to the effective strain at the front and release portion of the wave.

Mechanical testing of the specimens was carried out in the cylindrical geometry (6 mm diameter) under compression, and in the hat-shape geometry for obtaining high shear strains. The hat-shaped specimens were developed by Meyer and Manwarig [9]. Two different hat-shape sizes were used: 15 and 20 mm diameter, with corresponding heights of 5 and 15 mm. A Kolsky bar modified by Nemat-Nasser et al. [10] was used for the high-strain rate experiments, and an Instron uniaxial machine equipped with a furnace was used for quasi-static experiments.

Specimens for transmission electron microscopy were extracted from 3 mm cylinders taken from the high shear strain region by electro-discharge machining. They were sectioned in such a manner that the direction of shear is perpendicular to the plane of the foil. An electropolish solution containing 60% ortho-phosphoric and 40% water at 273 K provided the best results; in some cases, further thinning by ion-milling was necessary to produce electron-transparency in the shear region.

3. RESULTS

3.1 Dynamic Recrystallization at High and Low Strain Rates

Figure 1 shows the typical microstructure observed within the high strain region ($\gamma \sim 4$) of the hat-shaped specimens tested in the Hopkinson bar. This microstructure consists of small equiaxed grains (0.1-0.3 μm) in which the dislocation density is fairly low. Annealing (or recrystallization) twins were notably absent in these grains. It is possible to calculate the temperature rise within the shear region through the application of a constitutive equation that correctly represents the material behavior of the strain rate of

Figure 1. Microcrystalline structure characteristic of high-strain ($\gamma \sim 4$) high-strain-rate ($\dot{\gamma} \sim 4 \times 10^4 \text{ s}^{-1}$) deformation of copper.

interest; this strain rate is $\sim 5 \times 10^4 \text{ s}^{-1}$. The temperature range giving rise to the structure observed in Figure 1 is 500-600 K. Quasi-static (10^{-3} s^{-1}) tests carried out at different temperatures yielded flow stresses (at 30% plastic strain) that are shown in Figure 2(a). The experimental points were fit to a modified Johnson-Cook equation that is described elsewhere [11]. There is a drastic drop in the flow stress at a temperature of $\sim 570 \text{ K}$. Hat-shaped specimens identical to the ones used in the Hopkinson bar experiments were also tested at different temperatures at a strain rate of 10^{-3} s^{-1} and yielded the results shown in Figure 2(b). The specimen tested at 473 K shows a rather flat stress-displacement curve, whereas the specimens tested at 523 and 573 K display clear softening. This softening is indicative of dynamic recrystallization. At 673 K, on the other hand, the material work-hardens; thus, it is already in the recrystallized state, the effects of the shock deformation having been annealed out.

Transmission electron microscopy of the specimen tested at 673 K revealed a mixture of deformed and recrystallized grains, with varying dislocative densities. These features are revealed in Figure 3. Profuse annealing twin formation in the recrystallized grains is evident; this morphology is similar to the one observed by Mirklen et al. [12] and Wilbrandt and Haasan [13]. The large difference in the size of the recrystallized grains is a direct result of the strain rate differences between quasi-static and dynamic tests. Derby and Ashby [14] and Sandstrom and Lagneborg [15] predicted the following relationship between the steady-recrystallized grain size, d_{ss} , and strain rate $\dot{\epsilon}$:

$$d_{ss} = \frac{k}{\dot{\epsilon}^{1/2}}$$

Figure 2. (a) Flow stress vs. temperature for cylindrical specimens tested at 10^{-3} s^{-1} .
(b) Shear stress vs. displacement curves of hat-shaped specimens tested at high temperatures and a low strain rate (10^{-3} s^{-1}).

However, the fundamental morphological differences between the grains observed in Figures 1 and 3 suggest different mechanisms for dynamic recrystallization. Derby [16] classified dynamic recrystallization as a migration and a rotation mechanism. Whereas the recrystallized grains generated in quasi-static deformation involve the migration of boundaries, with the attendant generation of recrystallization twins; the microcrystalline structure of Fig. 1 is most probably generated by a rotational mechanism.

3.2 Effect of Grain Size on Shock and Post-Shock Response

The specimens with the four grain sizes given in Section 2 (9.5, 25, 117, and 315 μm) were subjected to an identical 50 GPa pulse and were subsequently tested. Figure 4 shows the significant difference in response. The curves on the left are for the as-annealed conditions and the curves on the right are translated by a strain equal to the effective strain imparted by shock (0.278). The shock response of the 315 μm specimen exceeded (by $\Delta\sigma$) that of conventionally work-hardened material. Thus, shock loading introduced a barrier density in the microstructure that exceeded that of quasi-static deformation. The 9.5 μm material, on the other hand, exhibited the opposite response. As expected, the annealed material is harder than the 315 μm material (Hall-Petch behavior). Shock loading was less effective than conventional plastic deformation. These differences are directly related to the grain-size dependence of

Figure 3. Small recrystallized grains ($d \sim 2\text{--}5\text{ }\mu\text{m}$) observed in the shear region of a hat-shaped specimen deformed at a strain rate of 10^{-3} s^{-1} and 673 K: A $\rightarrow$ curved (moving) boundaries; B $\rightarrow$ annealing twins.

Figure 4. Stress-strain curves of materials with (a) $9.5\text{ }\mu\text{m}$ and (b) $315\text{ }\mu\text{m}$, plotted with the origin offset by the approximate total transient shock strain.

Figure 5. Optical micrographs from shocked samples from experiment #2: (a) 9.5 μm , (b) 25 μm , (c) 117 μm , and (d) 315 μm ; microhardness numbers are indicated in the upper left hand corners.

Figure 6. Optical micrographs from the shear regions of hat-shaped specimens for (a) 9.5 μm and (b) 315 μm materials.

shock-induced mechanical twinning. Figure 5 shows the residual microstructures of the specimens with four grain sizes. Twinning is virtually absent for the 9.5 μm grain size and is profuse for the 315 μm specimen, showing consistent variation for the intermediate grain sizes. The strength differential $\Delta\sigma$ shown in Figure 4 can therefore be attributed to a dense network of twins. Indeed, the application of the Hall-Petch equation to the twin spacing ($\lambda=1\mu\text{m}$) with previously determined slope, yielded an increase in strength $\Delta\sigma=60\text{MPa}$, in fair agreement with $\Delta\sigma_2$.

This grain size dependence of shock response had a significant effect on shear localization in hat-shaped specimens. The large grain-sized material, exhibiting less work-hardening, localizes readily in the shock condition. In contrast, the 9.5 μm specimen, with a considerable work-hardening ability, does not localize in the hat-shape experiments. Figure 6 compares these two responses. The shear localization region is visibly thinner than the grain size for the 315 μm specimen. These differences might help to elucidate the observation of the grain size effect on the stability of jets in shaped charges. It should be noticed that Gourdin [17, 18] studied the effect of grain size on the high-strain-rate response of copper.

ACKNOWLEDGEMENTS

This research was funded by the U.S. Army Research Office University Research Initiative Program (contract #DAALO3-86-K-0169), by the National Science Foundation (grant #MSS-9021671) and by the Brazilian Government (U.R.A.). We thank Mr. F. Sandstrom, Prof. N.N. Thadhani, and Dr. J. Beatty for help with experiments. The dynamic tests were kindly performed by Mr. J. Isaacs.

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