Q. Powder Processing of Nanostructured Alloys Produced by Machining

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Objective

- Develop manufacturing technologies for creating advanced nanostructured alloys with enhanced mechanical properties for transportation applications.

Approach

- Create nanostructured chips by machining alloys of aluminum, titanium, and iron under controlled machining conditions.
- Characterize microstructure and mechanical properties of the nanostructured chips.
- Develop methods for converting the nanostructured chips into particulates suitable for processing into bulk forms.
- Develop powder processing methods, including shear-based densification (e.g., extrusion, rolling, forging) and infiltration processing, for creating monolithic and composite materials in bulk form with nanocrystalline microstructure.
- Characterize microstructure, mechanical properties, and performance of the bulk forms.

Accomplishments

- Produced nanostructured chips of a variety of alloys and metals—including Al6061-T6, copper, iron, commercially pure titanium, stainless steel and Inconel—by machining. The nanostructured chips are up to three times as hard as the bulk material.
- Achieved typical grain sizes in the chips of 50–700 nm, depending on the strain and temperature induced by the machining.
- Consolidated cylindrical bulk samples of nanostructured Al-6061T6/pure aluminum composites by powder extrusion. Densification in excess of 95% has been obtained while retaining the hardness of the nanocrystalline structures.
- Fabricated bulk samples of nanostructured Al-6061T6, with small amount of polymer as binder, by infiltration and pressing.

Future Direction

- Continue development of shear-based densification and infiltration methods to produce bulk monolithic and/or composite alloy samples with nanocrystalline microstructure, using the chips as precursor materials.
- Characterize microstructure and properties of the bulk forms to demonstrate retention of the nanostructures and enhanced mechanical properties.
- Determine tensile strength, ductility and density of the bulk forms to demonstrate integrity of the consolidated alloy samples.
- Identify 1–2 applications in the transportation sector for prototype development.

Introduction

It has been widely reported that nanostructured materials, composed of nanometer-scale grains, have properties significantly different from those of conventional materials.[1,2] These properties include higher hardness, strength and ductility, and superplasticity at relatively low temperatures and sintering temperatures that are several hundred degrees below those of microcrystalline powders.[3,4] Widespread use of these materials, including bulk forms (components and solid bodies) with nanocrystalline microstructure, has been sought; however, the cost of creating these materials, frequently quoted as being more than $100 per pound, and the inability to produce them in tonnage quantities with reproducible properties have restricted their broad application.[5]

This project builds on the recent discovery of a low-cost means of producing nanostructured materials in essentially any alloy.[6,7] The discovery that chips produced during normal machining operations are composed entirely of nanocrystalline structures of high hardness (strength) provides a low-cost, direct way of making these materials in large volumes. Comminution of the nanostructured chips will enable large-scale production of nanocrystalline particulate, which can be converted into nanostructured bulk forms using powder processing methods. Alternatively, the chips and particulates may be used as continuous or discontinuous reinforcements in metal or polymer matrices to create advanced composite bulk forms.

Approach

Chips were produced by machining, ranging from soft alloys [e.g., oxygen-free high-conductivity (OFHC) copper, aluminum 6061-T6[to high-strength alloys (e.g., Inconel, 52100 steel); and the microstructure and hardness of the chip and bulk (workpiece) material was characterized.[7,8] The bulk materials were annealed in an inert atmosphere before machining so that he initial grain size in all cases was greater than 20 μm. Typical dimensions of chip samples examined were 100–3000 μm width, 100–1000 μm thickness, and at least 5 mm length, with the smaller chips coming from the higher-strength steels. The machining conditions were selected so that the temperature increase in the deformation zone was minimal for all materials.

Transmission electron microscopy (TEM) was used to characterize the nanoscale defect structures in the metal chips and to measure grain size, grain size distribution, and grain misorientation. Specimens for TEM examination were prepared from chips produced under well-defined deformation conditions (strain, strain rate, temperature) by standard mechanical, ion beam, and electrochemical thinning techniques. Bright-field imaging coupled with electron diffraction analysis was conducted in a scanning TEM at Purdue and at the High Temperature Materials Laboratory (HTML) at Oak Ridge National Laboratory (ORNL). The hardness of the chip and bulk samples was measured by Vickers and nano-indentation. Uni-axial tests were carried out on miniature copper tension samples prepared from the chips.

Preparation of bulk forms from nanostructured particulate derived from the machining chips was initiated using powder extrusion and infiltration processing. For powder extrusion, nanostructured Al6061-chips were converted into particulate using attrition milling. The resulting particulate was mixed with varying weight percentages of commercially available, gas-atomized aluminum powder; this mixture was powder-extruded through steel dies at extrusion ratios of 10–60. The extruded samples were prepared in the form of cylinders of up to 10 mm in diameter and 5–25 mm in length.

Results

Figure 1 shows a TEM image of a typical Al 6061-T6 chip. Shown in the inset is the corresponding selected area diffraction pattern, typical of a polycrystalline structure. The grain size is seen to be in the range of 50–80 nm. Misorientation measurements carried out using convergent beam electron diffraction in the TEM have established that more than 50% of the grain boundaries in the Al 6061-T6 chips are large-angle; the fraction of the large-angle boundaries is greater in chips produced at higher levels of strain. Evidence for mechanical twinning has been seen in some of the highly strained chips, akin to that reported elsewhere.[9] Similar nanocrystalline structures have been observed in a variety of alloys, including high-strength 52100 and M2 steels (Figure 2). Ultrafine sub-grain structures, nanoscale grains, and dynamic recrystallization have been demonstrated in a variety of materials by varying the conditions of strain and temperature during chip formation. An example of such nanostructural changes can be seen in the sequence of TEM pictures in Figure 3 of OFHC copper chip samples produced with different levels of shear strain (~ 3–20). A *switchover* from sub-grain to nanoscale, equiaxed grain structures at the higher levels of strain (Figure 3) appears to be a consequence of the onset of dynamic recrystallization at a strain of ~ 13. This switchover, which appears controllable in different materials by varying the deformation conditions, has not been realized in a single stage of deformation in any of the frequently studied severe plastic deformation processes. These observations suggest that machining also offers a unique framework in which to systematically explore the formation of nanocrystalline structures by large strain deformation.

Table 1 gives a summary of the hardness values measured on the chips and the bulk samples. The chips are seen to be up to three times as hard as the corresponding bulk samples prior to machining. The hardness of the 52100 steel chip is 1310 kg/mm^2, which is comparable to that of patented steel wire, one of the hardest and strongest steel structures known.[10] As has been reported in many studies,[1–4] nanostructured alloys have significantly greater strength than their microcrystalline counterparts. Uni-axial tensile tests carried out on miniature

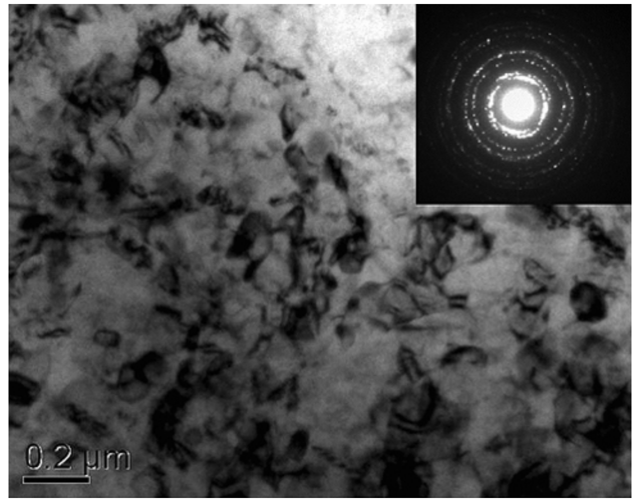

Figure 1. Nanostructured Al 6061-T6

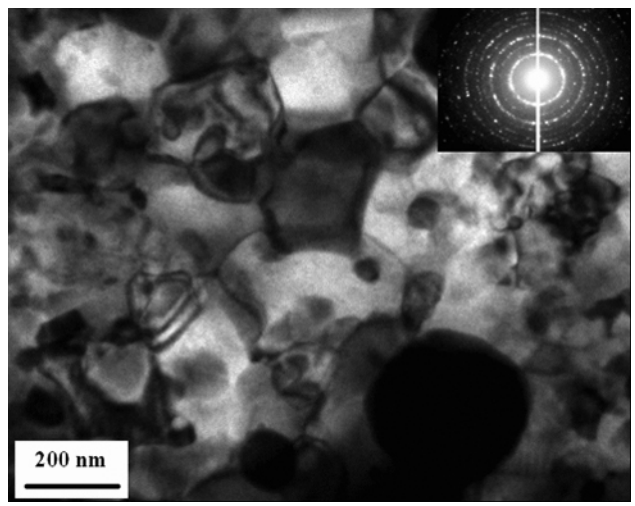

Figure 2. 52100 steel showing nanoscale ferrite grains and carbide particles.

OFHC copper chip samples have shown the increases in strength to parallel those in hardness. These increases in chip strength and hardness are much higher than those that result from extrusion, forging, and rolling.[11]

Figure 4 shows an optical micrograph of a partially formed OFHC copper chip, as yet not separated from the bulk.[6] Superimposed on the micrograph are Vickers hardness (strength) values recorded at different locations. The hardness shows a steep increase over a very narrow deformation zone between the chip and the bulk. This zone, where chip formation occurs, is characterized by very large deformations, as highlighted by the flow lines in the chip in Figure 4. While grains are clearly

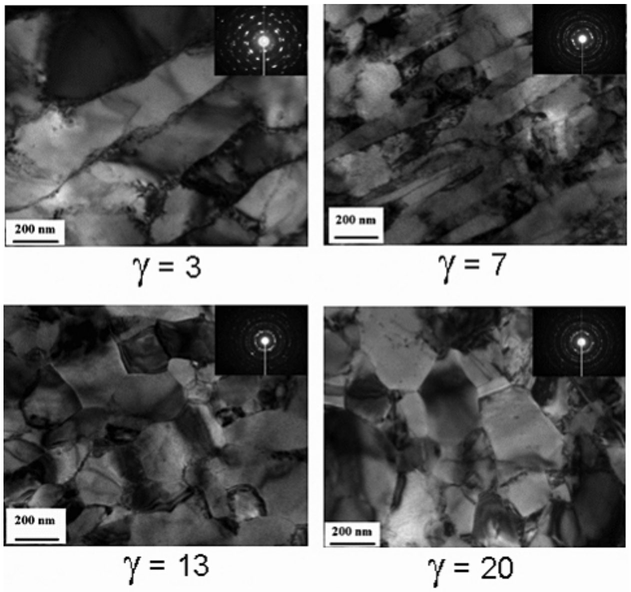

Figure 3. Microstructure of OFHC copper chips at different levels of shear strain (γ). Features typical of onset of dynamic recrystallization are visible in the chip produced with a shear strain of ~13.

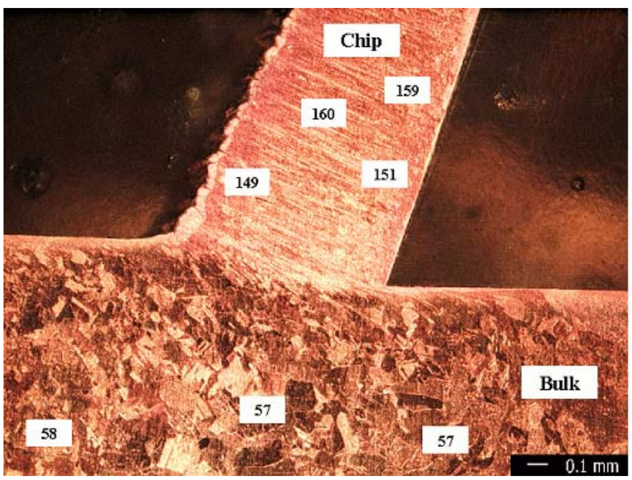

Figure 4. Partially detached OFHC copper chip. Note large hardness increase across shear plane.

visible in the bulk OFHC copper sample, no grains can be resolved in the chip or near the deformation zone, consistent with TEM observations that the microstructure is nanoscale.

Figure 5a shows a cylindrical sample of Al6061-T6/Al composite produced by powder extrusion. This composite is composed of 50% by weight of nanostructured 6061 chip particulate blended with an equal weight of aluminum powder. Density measurements showed the sample to be consolidated to 97% of the theoretical density. Figure 5b shows a transverse cross-section of this sample; the porosity is seen to be negligible, consistent with the measured density. Figure 5c shows the distribution of hardness over the cross-section. The higher hardness values of ~160 VHN are similar to those measured in the nanostructured chip particulate, suggesting that the nanostructure is retained. The remainder most likely correspond to that of the aluminum powder strain-hardened by the extrusion. The early observations pertaining to hardness, density, and microstructure, made on the powder-extruded samples, are promising vis-à-vis densification and nanostructure retention.

We have also been successful recently at producing bulk Al6061-T6 samples from nanostructured chip particulate via an infiltration/pressing approach using a small amount (<10%) of epoxy as binder.

Conclusions

Nanostructured materials with grain sizes as small as 70 nm have been produced by controlled 2-dimensional machining in a wide variety of metals and alloys. The hardness of these alloys is up to three times that of their micro-crystalline counterparts. Preliminary results on consolidation of these materials using shear-based densification and infiltration processes are promising. A variety of low-temperature consolidation protocols have been identified for producing bulk forms using these nanostructured chips as precursor materials. These include powder extrusion, rolling, and forging; infiltration processing; dynamic compaction; rapid prototyping; and spraying. In the next year, intensive efforts are planned on using these processes to address the grand challenge of consolidating these nanostructured materials into high-performance bulk forms. Property and performance studies will be initiated on these consolidated samples to determine indices of relevance to transportation applications. Close collaboration with the HTML is envisioned on various aspects of this work.

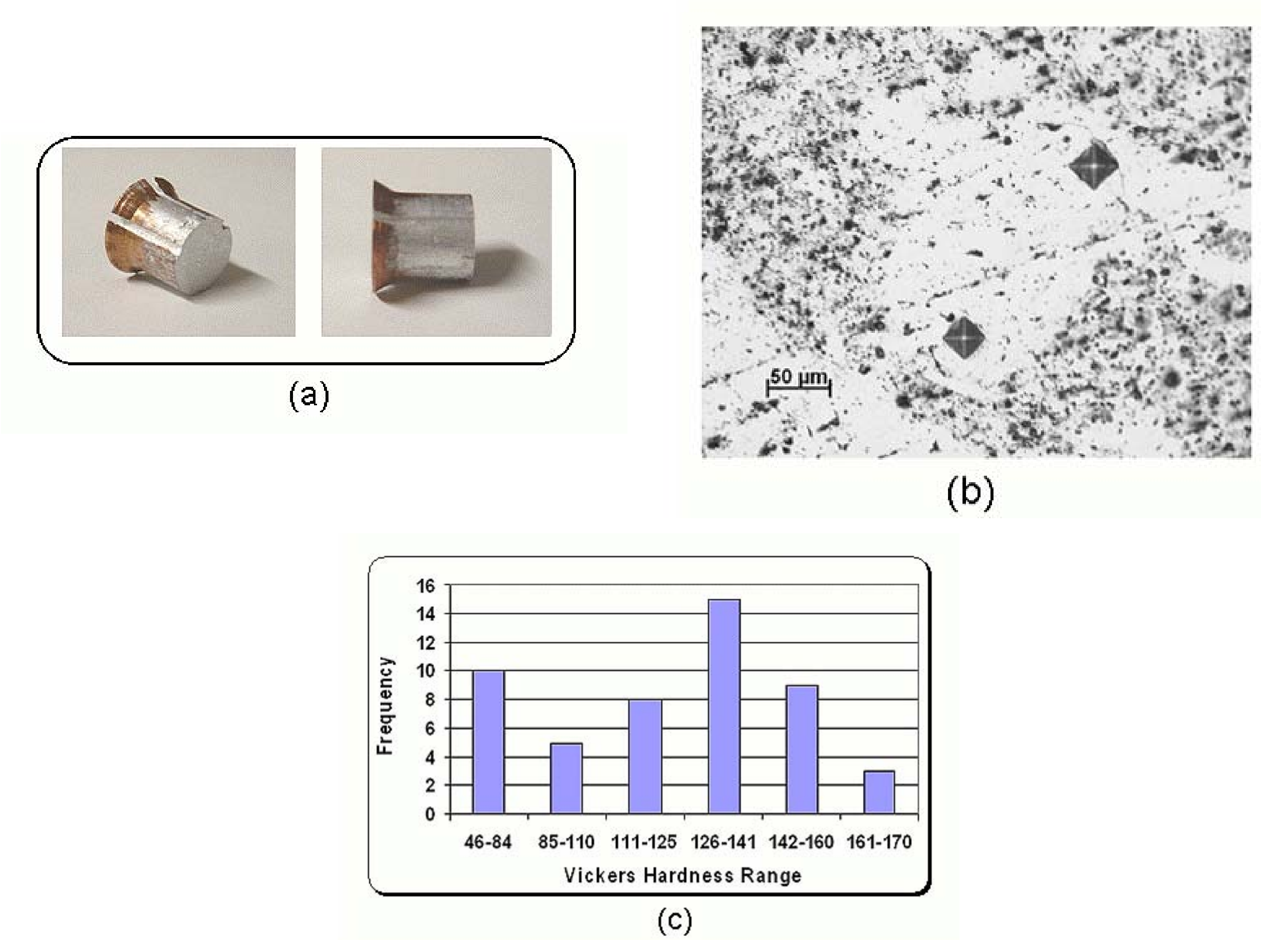

Figure 5. Characteristics of powder-extruded Al-6061T6/Al composite: (a) typical extruded samples, (b) microstructure of a transverse cross section, and (c) hardness distribution determined by Vickers indentation.

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