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 determine relationships between structure and mechanical properties of the nanostructured chips.
- Develop methods for converting the nanostructured chips into particulates suitable for processing bulk forms.
- Develop low-temperature consolidation routes for creating monolithic and composite materials without significant coarsening of the nanocrystalline microstructures.
- 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.
- Developed low-temperature consolidation routes that limit coarsening in Al6061-T6, and steels. Achieved a high degree of interparticle bonding in room temperature powder extrusion of nanostructured Al6061-T6.
- Developed highly stable nanostructured (HSN) materials, for example, Inconel 718, Waspaloy, of high-strength through a combination of grain-size refinement by machining and creation of fine-scale precipitates by heat treatment.
- Developed a new process, extrusion-machining, to engineer bulk samples, for example, foils, filaments, with ultra-fine grained (UFG) microstructure and enhanced mechanical properties.

Future Direction

- Continue development of low-temperature consolidation routes to produce bulk monolithic and/or composite alloys with nanocrystalline microstructure.
- Characterize microstructure and properties of the bulk forms to demonstrate retention of nanostructures and enhanced mechanical properties.
- Determine tensile and wear properties of bulk forms to demonstrate integrity of the consolidated alloy samples and guide application.
- Develop (identify 1–2) applications in the transportation sector for prototype development.

Introduction

This project builds on the recent discovery of a low-cost means of producing nanostructured materials in essentially any alloy via normal machining. Comminution of the nanostructured chips enables large-scale production of nanocrystalline particulate, which can be converted into nanostructured bulk forms using powder processing methods. Particulate having controlled size and shape can also be produced by a modulated machining process. The chips and particulates also may be used as continuous or discontinuous reinforcements in metal or polymer matrices to create advanced composites.

Progress from FY 2005 in two main areas is highlighted in this report: (1) low-temperature consolidation routes, primarily of ultrafine-grained Al6061-T6 and (2) microstructure development in machining of solution-treated alloys. Modulated machining could be in there as a third, but not necessary or enough space.

Approach

Three routes for consolidation and densification of the nanostructured particulate to produce monolithic and composite materials were pursued: (1) shear-based deformation processing (e.g., powder extrusion); (2) rapid infiltration processing using low-melting metal alloys; and (3) polymer die-press bonding.

These routes are all geared toward densification and bonding of the particulate while minimizing thermal coarsening. For the powder extrusion, nanostructured Al-6061-T6 chips were converted into particulate using attrition milling. The resulting particulate was mixed with varying weight percentages of commercial 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 up to 10 mm in diameter and 5–25 mm in length.

The polymer bonding is a new approach in which a thermosetting polymer resin is used, together with high-pressure compression molding in a steel die to yield a structure consisting of low-volume fractions of polymer bonding the nanostructured metal particles. The focus so far has been on Al-6061-T6 chip powders bonded with different epoxies. This approach is based on the observation that the cure conditions of the resin are well below the known time-temperature conditions for coarsening (and loss of hardness) of the nanostructures in the chip particles. Application for lightweight, wear-resistant components is envisioned.

Another avenue of investigation has focused on creation of high-strength, high-temperature nanostructured materials such as titanium and nickel-based alloys, with exceptional thermal stability of the microstructure. In this context, we have demonstrated a means of extending the temperature range for structural applications of nanomaterials through the introduction of a dispersion of second-phase precipitates, whose thermal stability determines the temperature at which the strength deteriorates. Precipitate-stabilized nanostructured materials synthesized from solution-treated Inconel 718 are shown to be extremely stable even after prolonged heat treatment for 500 h at temperatures as high as one-half of the melting point; we designate them as "highly-stable nanostructured (HSN) materials." The enhanced thermal stability of the material's strength results from retention of both the fine microstructure and the fine precipitate phase coexisting at the high temperatures. This novel microstructure was created by first deforming solution-treated Inconel 718 to a

large shear strain by machining, followed by thermal aging of the chip to create fine-scale precipitates.

Results

Room Temperature Powder Extrusion of Al-6061-T6 Chip Particulate

Extrusion of nanostructured Al6061-T6 chip particulate results in poor densification and bonding. However, by blending the chip particulate with pure aluminum powder, near complete densification is possible. The effect of chip powder particle size relative to the pure aluminum (Ampal, 99.9%, gas-atomized, ~60 μm) and extrusion ratio are under investigation. Figure 1 shows the extrudate and corresponding microstructure for a ~180-μm nano-chip particulate blended 50–50 with pure aluminum and extruded at room temperature with an extrusion ratio of 42. Immersion density measurements indicated a relative density of 98.7%, consistent with the small amount of porosity observed in optical microscopy. The chip particles are clearly distinguished in the micrographs and appear well—bonding with the continuous pure aluminum matrix. Tensile testing (3-mm-diam gage sections) completed late this year confirmed a significant degree of bonding. A fully sintered pure aluminum powder specimen was tested in the same manner to serve as a control sample. The detailed test results are as follows:

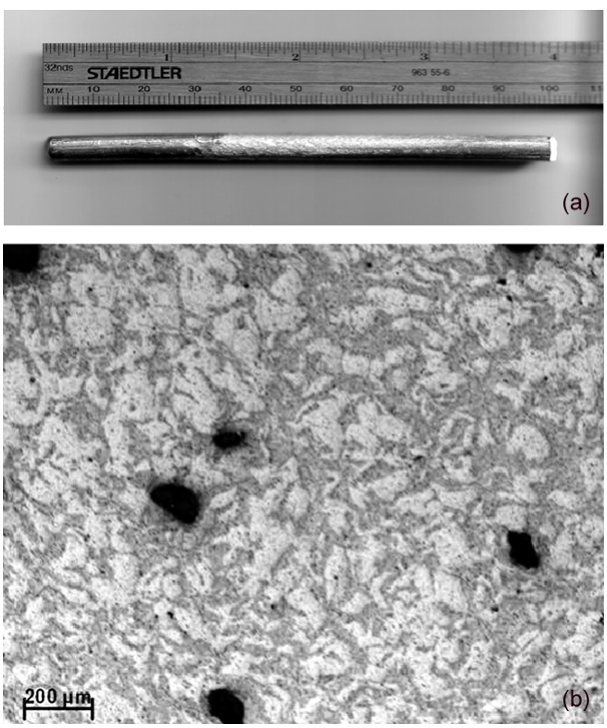

Figure 1. (a) Macrograph showing Al-6061-T6 nanochip/pure aluminum (1:1 by volume) extrudate; extrusion ratio = 42. (b) Optical micrograph of corresponding microstructure in cross-section showing small amount of residual porosity on the order of the nanochip particle size but otherwise well-bonded structure.

Material	Properties (YS/UTS/%El)
Fully sintered pure aluminum	60 MPa/75 MPa/5%
6061 nanochips + pure aluminum	170 MPa/170 MPa/nil
6061 nanochips + 601AB	200 MPa/200 MPa/nil

The measured strength values indicate significant interparticle bonding in the ambient temperature extruded materials based on machining chips. The tensile strength values furthermore support the apparent interparticle bonding revealed by metallography, as we have reported before. For comparison, sintered 601AB exhibits tensile property sets from 50 MPa/125MPa/8% to 240 MPa/250 MPa/2%, depending on the density and temper.[1] Work is ongoing to quantify the degree of bonding in terms of tensile strength, which, with such limited ductility, is also strongly dependent on the size of pores and other flaws.

Larger scale extrusions using the ORNL extrusion facilities are in progress.

Polymer-Bonded Chip Particulate Composites

Die press bonding has been used to produce bulk Al-6061-T6 samples from nanostructured chip particulate using a small amount (<10%) of epoxy as binder. Figure 2 shows a typical example. Small amounts of porosity persist, especially where larger particles were not packed uniformly, but most particle boundaries show a thin bond layer of epoxy. Epoxy and porosity volumes as low as 6% have been achieved so far, and preliminary nanohardness measurements show little or no loss of hardening imparted in producing the chips, consistent with separate measurements of coarsening-softening kinetics in this alloy.

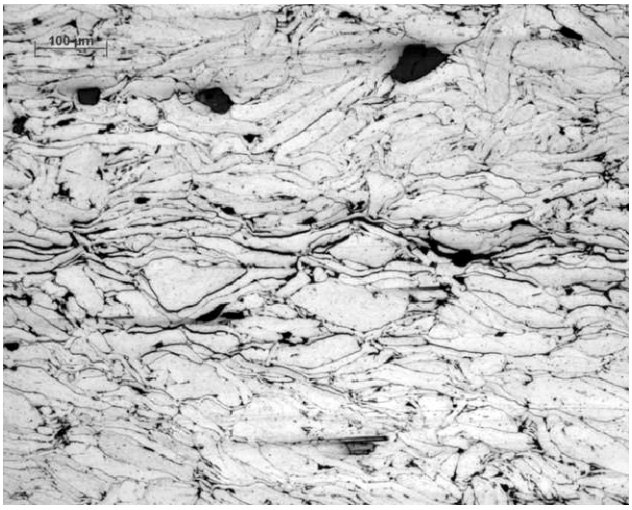

Figure 2. Optical micrograph showing epoxy-bonded Al-6061-T6 chip particulate (15–100 µm) pressed at 1000 MPa and cured at 100°C.

Work in the next year will proceed to tensile testing in order to evaluate the degree of bonding. Although high strength and ductility are not predicted, the high hardness should impart improved wear resistance together with the shaping advantages of powder processing.

Highly Stable Nanostructured Materials

Figure 3 illustrates the nanostructured material composed of grains typically 100 nm in size produced from solution-treated microcrystalline Inconel 718 (grain size ~50 µm in the initial state) due to plastic shear strains of ~6. The microstructure is composed primarily of equi-axed grains, along with some elongated structures. The diffraction pattern inset in Figure 3 indicates the presence of large angle grain boundaries in this nanostructured material. The Vickers microhardness value of this microstructure was 550 kg/mm^2 (~5.4 GPa), nearly twice that of the undeformed bulk microcrystalline solution-treated material (300 kg/mm^2).

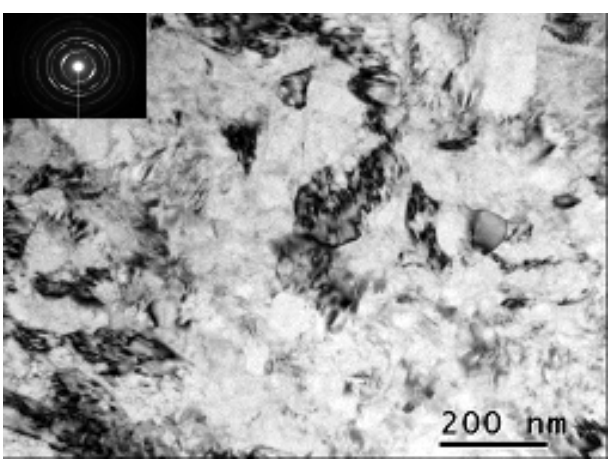

Figure 3. TEM micrograph of nanostructured Inconel 718 produced by large strain machining (shear strain ~6) from solution-treated microcrystalline Inconel 718. Average grain size is ~100 nm.

When this nanostructured material was heat treated at 600°C (~ one-half the melting point), a rapid hardening was found that can be associated with precipitation in the nanostructured solution-treated material. Figure 4 illustrates the variation in microhardness of the nanostructured Inconel 718 with temperature and the attainment of a peak hardness value of 630 kg/mm^2 (~6.2 GPa) after about 6 h of annealing. This hardness value corresponds to a tensile yield strength of ~2 GPa for this material. Figure 4 confirms the extraordinary stability of material strength (hardness) of this HSN Inconel 718 even after 30 h at 600°C (~ one-half of the melting temperature). The peak hardness of

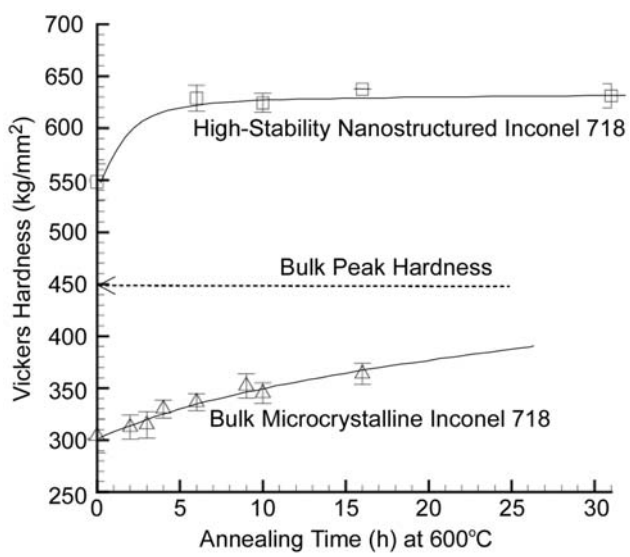

Figure 4. Variation of Vickers hardness with annealing time at 600°C for high-stability nanostructured (HSN) Inconel 718 and bulk microcrystalline Inconel 718 (undeformed bulk, grain size ~50 µm). The bulk peak hardness of 450 kg/mm^2 is that of microcrystalline Inconel 718 after prolonged double-ageing treatment and is shown for comparison. HSN Inconel 718 was also heat treated at 600°C for 500 h, and its hardness was found to remain stable at ~630 kg/mm^2.

630 kg/mm^2 is unchanged throughout this heat treatment, and this hardness value is still ~40% greater than that of peak-aged Inconel 718 (~450kg/mm^2) in the conventional microcrystalline form. We also performed annealing experiments at 600°C for 500 h on the HSN Inconel 718, and even such prolonged heat treatment was found to have a negligible effect on the stable hardness value (~630 kg/mm^2).

The materials design principle propounded above can be generalized to other precipitation-treatable materials and offer a new line of development for this important class of structural materials. Other material systems that form stable, fine precipitate phases, for example, P-series stainless steels, Waspaloy, can be expected to behave in a similar manner in the nanostructured state. Exceptional thermal stability and retention of the material strength are ensured by the stable, fine precipitate phase and the fine microstructure coexisting at high temperatures for significant lengths of time, resulting in a class of HSN materials with enormous potential for high-strength and/or high-temperature applications. These avenues will be investigated in detail in the coming year.

Nanostructured Pure Titanium by Machining

Figure 5 shows a TEM micrograph and selected area diffraction pattern of nanostructured titanium formed in machining chips. The starting grain size of the commercially pure titanium was ~50 µm, and TEM revealed a grain size range of 50 to 200 nm in the chip specimens. Correspondingly, Vickers hardness increased from 144 ± 5 to 230 ± 9 kg/mm^2, consistent with the machining response of a wide range of other pure metals and alloys.

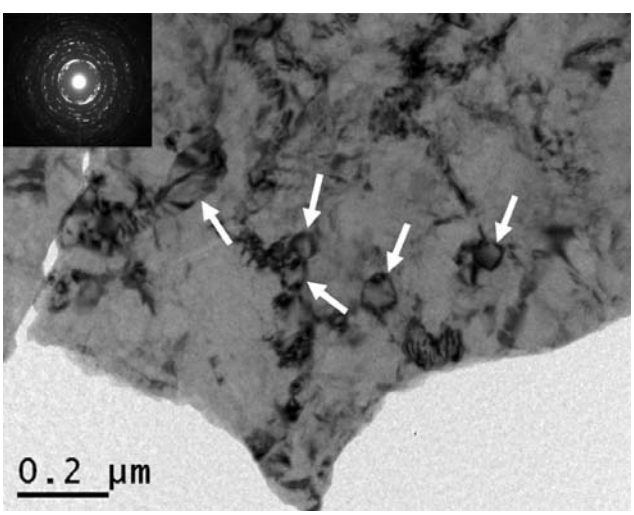

Figure 5. TEM micrograph.

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Patents

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