ABSTRACT

The creation of nanostructured materials with enhanced mechanical properties by controlled chip formation has been demonstrated. The present study examines the microstructure and mechanical properties of chips from various alloys - Waspaloy AMS 5704, Inconel 718, Al 6061-T6, and titanium - produced in aerospace machining operations. While the deformation conditions with respect to chip formation may be 'less than controlled' in these cases, it is nevertheless seen that the chips created are composed entirely of nanocrystalline structures of high hardness and strength. The microstructural characteristics and properties of these chips are compared and contrasted with those produced under controlled conditions of strain and temperature. The results suggest that aerospace machining chips can be up-scaled as high-performance structural materials with substantial cost benefits.

INTRODUCTION

It has been widely reported that nanostructured materials, composed of sub-micrometer scale grains, have properties significantly different from those of conventional materials [1, 2]. These properties include higher hardness, strength and ductility, 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 in excess of one hundred dollars per pound, has restricted their broad application [5].

The present study seeks to exploit the discovery of a low-cost means of producing nanostructured materials in essentially any metal or alloy [6, 7]. Figure 1 is a transmission electron microscope (TEM) micrograph of a chip created from Al 6061-T6 aluminum alloy (initial grain size: 75 µm) in a controlled plane-strain machining operation using a tool of rake angle -20°. The speed of the machining was kept sufficiently low so as to minimize any temperature rise in the chip formation zone. The microstructure is seen to be very fine-scale and comprised of equi-axed grains with a mean size of ~80 nm. Furthermore, the Vickers hardness of the chip was ~30% greater than that of the bulk material with a microcrystalline structure. From measurements of the deformed and undeformed chip thickness, and the tool rake angle, the shear strain imposed in this chip during its formation was estimated to be 5.2. The nano-scale microstructure of the chip is a consequence of the material being subjected to very large strain deformation during chip formation [6, 7]. Nanostructured materials, with hardness up to three times that of the bulk material, have been created in a variety of metals and alloys using this machining approach.

FIG. 1 Bright field TEM micrograph of Al 6061-T6 chip machined under laboratory conditions. The shear strain in the chip is 5.2 and the grain size is ~ 80 nm.

The discovery that chips produced during controlled laboratory machining operations are composed entirely of nanocrystalline structures of high hardness (strength) suggests low-cost, direct ways of making these materials in high volume. One of these is the exciting possibility that chips produced in aerospace machining operations usually disposed as low-volume scrap, are endowed with a nanocrystalline or an ultra-fine grained (UFG) microstructure. Should this be the case, then a very low-cost and abundant source of...
advanced materials will be shown exist within the aerospace manufacturing sector. That this is the case is by no means obvious since chips in industrial machining operations may be created under less than "controlled conditions". For example, the machining speeds in industry are significantly higher than those used in the laboratory plane-strain machining cited above. This could cause recrystallization and grain growth during chip formation resulting in a chip microstructure composed of large micrometer-sized grains. But it is also likely that a fine scale microstructure is preserved in the chip, since during its formation, the chip is subjected to elevated temperatures only for a relatively short duration of time [8]. The present study was undertaken with the objective of examining the microstructure and hardness of chips from aerospace alloys machined in industry.

The aerospace sector includes machining advanced metals and alloys for structural performance and durability, resulting in a significant production of machined chips that are typically recycled or disposed. The buy-to-fly ratio (ratio of weight of metal purchased to the weight of the finished parts) is a measure of the amount of scrap generated in manufacturing. Depending on the part being made, these ratios can range from less than 5:1 to greater than 20:1 [9]. For example, the main landing gear fitting in an Airbus A340 is machined from a 7.5 ton aluminum casting, and at the end of the process landing gear fitting in an Airbus A340 is machined from a 7.5 ton aluminum casting. For this purpose, chip samples and control samples from the bulk material were prepared either by an electrolytic jet thinning technique or by a wedge-polishing technique. In the electrolytic jet thinning technique, the samples were first ground to a thickness of ~100µm using an abrasive polishing wheel. Three millimeter diameter disks were then punched out of the ground samples and made electron transparent by electrolytic twin-jet thinning on a Struers Tenupol-5 polishing system. For the wedge-polishing technique, the specimens were mechanically thinned by abrasive polishing to form shallow wedges. Each wedge was then mounted on a copper slot grid and ion milled for a short duration of time (~5-10 minutes) in a Gatan Model 600 dual ion mill to create an electron transparent specimen. Care was taken to avoid any prolonged heating of the specimens during the mechanical thinning and ion milling in order to ensure the integrity of the microstructure of the chip.

Bright- and dark-field images, along with selected area diffraction patterns (SAD), were taken to analyze grain size, defect structure, and subgrain misorientation. The average grain size was determined using Heyns intercept method [11] by examining 20-50 grains. If the aspect ratio of the grains (defined as the ratio of the length to width of a grain) was greater than 2, then the grain was considered as elongated; otherwise the grain was taken to be equi-axed. For those chips with elongated grain microstructures, the mean width of a grain is reported as the grain size.

Some of the chip and bulk samples were mounted in a thermosetting resin and polished for metallographic observations. The finely polished samples were used for microstructure analysis and hardness testing. Hardness (strength) measurements were made using Vickers indentation taking care to ensure that the size of an indentation, as measured by its diagonal length, was kept about the same in the bulk and chip samples so as to minimize any uncertainties arising from an indentation ‘size effect’. An average value for the hardness was obtained by performing at least 20 indents on each sample.


ewperimental Procedure

Chips of a variety of commercially pure metals and alloys, viz., titanium, Inconel 718 (solution-treated), Waspaloy AMS 5704 and Al6061-T6, were collected from machining operations carried out in the aerospace sector. In all cases, the initial grain size of the bulk material from which the chips were created was in excess of 30 µm. The conditions of machining, while unknown, may be taken as what is typically used in aerospace manufacturing. The objectives were to determine if the chips were nanostructured, and what, if any, enhancements had occurred in their mechanical properties as a consequence of the machining-induced deformation. For reference purposes, chips from some of these alloys were also created under controlled laboratory machining conditions in which the strains imposed in the chip were known.

The microstructure of the chips and some of the bulk specimens was characterized using a JEOL 2000FX Transmission Electron Microscope (TEM) operating at 200 kV. For this purpose, chip samples and control samples from the bulk material were prepared either by an electrolytic jet thinning technique or by a wedge-polishing technique. In the electrolytic jet thinning technique, the samples were first ground to a thickness of ~100µm using an abrasive polishing wheel. Three millimeter diameter disks were then punched out of the ground samples and made electron transparent by electrolytic twin-jet thinning on a Struers Tenupol-5 polishing system. For the wedge-polishing technique, the specimens were mechanically thinned by abrasive polishing to form shallow wedges. Each wedge was then mounted on a copper slot grid and ion milled for a short duration of time (~5-10 minutes) in a Gatan Model 600 dual ion mill to create an electron transparent specimen. Care was taken to avoid any prolonged heating of the specimens during the mechanical thinning and ion milling in order to ensure the integrity of the microstructure of the chip.

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RESULTS

Figure 2 shows an optical micrograph of an etched Al 6061-T6 chip from an aerospace machining operation. The microstructure is seen to be composed of flow lines characteristic of large strain deformation. No grains could be resolved under the optical microscope indicating that the grain size in the chips is on the order of 1 µm or less; however, a direct assessment of the microstructure could be made with the TEM. Similar flow-line type microstructures were seen in Al 6061-T6 chips obtained from other industry sectors.

Figures 3a) and 3b) are TEM micrographs of two Al 6061-T6 chip samples. The figures show an elongated subgrain type microstructure with the mean subgrain size in the width direction being ~ 180 nm in Figure 3a) and ~ 100 nm in Figure 3b). The inset SAD patterns in the figures suggest a low misorientation
between the subgrains in Figure 3a) and larger misorientation angles in Figure 3b); this latter inference is based on the more smeared SAD pattern of Figure 3b). The nanocrystalline microstructure of Figure 3b) is similar to that of Figure 1, which is the microstructure of an Al 6061-T6 chip, with a grain size of 80 nm and composed of highly misoriented grains, produced under laboratory conditions. The Vickers hardness of the Al 6061-T6 chips is 135-140 kg/mm², which is about 30% greater than that of the bulk Al 6061-T6, see Table 1. These chip hardness values are somewhat lower than the highest hardness of ~155 kg/mm² measured in the Al 6061-T6 chip created in the laboratory.

Significant refinement of microstructure and enhancements in hardness were also observed in chips created in machining of Waspaloy, Titanium and Inconel 718. Figures 4a) and 4b) are TEM micrographs from different regions of a Waspaloy chip. The microstructure is seen to be composed of elongated subgrains in one region (Figure 4a)) and of equi-axed sub-100 nm grains in another region (Figure 4b)).

FIG. 2 Optical micrograph of microstructure of an Al 6061-T6 chip from an aerospace machining operation. A “flow line” type microstructure, characteristic of large strain deformation, is seen.

FIG. 3 TEM micrographs of Al 6061-T6 chips. The microstructure in (a) is composed of subgrains with a mean width of ~180 nm. The microstructure in (b) is composed of somewhat equi-axed grains with a mean size of ~100 nm and with larger values of misorientation angles between the grains. Insets show the selected area diffraction (SAD) patterns.
FIG. 4 Bright field TEM images of two different regions of a Waspaloy chip showing elongated nanostructures in (a) and equi-axed nanostructures in (b) with subgrain sizes in the range of 50 – 100 nm.

<table>
<thead>
<tr>
<th>Material</th>
<th>Sample</th>
<th>Hardness (kg/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al 6061-T6</td>
<td>Bulk</td>
<td>110</td>
</tr>
<tr>
<td></td>
<td>Chip</td>
<td>135-140</td>
</tr>
<tr>
<td>Waspaloy</td>
<td>Bulk</td>
<td>390</td>
</tr>
<tr>
<td>AMS 5704</td>
<td>Chip</td>
<td>605</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>Bulk (solution-treated)</td>
<td>280</td>
</tr>
<tr>
<td></td>
<td>Chip</td>
<td>570</td>
</tr>
<tr>
<td>Titanium</td>
<td>Bulk</td>
<td>145</td>
</tr>
<tr>
<td></td>
<td>Chip</td>
<td>230</td>
</tr>
</tbody>
</table>

TABLE 1. Vickers hardness of the bulk and chip samples

FIG. 5 Bright field TEM image of a titanium chip with the selected area diffraction (SAD) pattern in inset. The grains are 50 – 200 nm in size.

Consolidation of nanocrystalline chips to fabricate bulk forms by conventional powder metallurgy techniques is likely to involve some heating. Thus, thermal stability of the nanocrystalline microstructure is a necessary characteristic for retention of the superior mechanical properties in the consolidated bulk. A recent study of the annealing behavior of nanocrystalline Al 6061-T6 chips, created under laboratory conditions, identified time-temperature processing windows wherein there was minimal coarsening and loss of hardness of the nanocrystalline microstructures [12].

A preliminary study was conducted to assess the annealing behavior of the nanocrystalline Al 6061-T6 chips produced in aerospace machining operations. The data from the annealing study, which is shown in Figure 6, demonstrates the variation in hardness with annealing time at 175°C, the peak ageing temperature for Al 6061-T6. The data confirms that both samples of the chips from the aerospace operations show a similar variation in hardness with annealing time. There is an initial rapid decline in hardness followed by a period in which the decline in hardness is much more gradual. Furthermore, their annealing behavior is very similar to that of the Al 6061-T6 chips produced under the laboratory machining conditions as suggested by the data shown in Figure 6. TEM observations of the laboratory chips after different durations of annealing showed that the initial rapid decline in hardness is associated with a coarsening of the microstructure. But even after one hour of annealing, the grain size was still in the sub-micron range. While we
did not study the grain size changes in the chips produced in the industrial operations, it is our inference based on the hardness versus annealing time observations that the grain size changes are similar to those occurring in the laboratory chips. These observations suggest that warm consolidation of Al 6061-T6 chips, be they from the industrial sector or from the laboratory, could be carried out around 175°C with minimal loss of strength.

![Graph](image)

**FIG. 6** Variation of Vickers hardness with annealing time for Al 6061-T6 chips. Samples 1 and 2 are from two separate batches of industrial chips.

**CONCLUSION**

A study has been made of the microstructure and hardness of chips created in machining of aerospace alloys from various industrial machining operations. The microstructure in all cases is seen to be nanocrystalline and the chip materials are much harder than the bulk material. While the scale of the microstructure in these chips is, likely, somewhat coarser than that of the chips created under laboratory conditions with controlled shear strain, the observations clearly show significant refinement of the microstructure vis-à-vis the bulk material. Perhaps, more importantly, the results show that chips from industrial machining operations offer an abundant source of nanocrystalline materials with enhanced mechanical properties.

Although nanostructured materials exhibit novel intrinsic property combinations, achieving this potential in bulk forms, i.e., component and product applications, has been hampered by their high cost [5]. The present study has shown that a continuous supply of nanostructured materials may be obtained from industrial machining chips. Nanostructured materials derived from machining chips, in combination with powder metallurgy, spraying and composite processing methods, offer the potential for creating advanced materials with new and interesting combinations of properties. These are likely to be extremely attractive for applications in the aerospace, ground transportation, and bio-medical sectors where weight reduction and enhanced performance are critical.

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