Microstructural analysis of nanostructured aluminum alloy strips created from machining based deformation process

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Abstract

Aim of present research endeavour is to fabricate nanostructured strips of aluminium alloy by a machining based deformation process. Aluminium 6061 was used as a test material. Furthermore, X-Ray diffraction (XRD) is used for deformation analysis of strips to check the level of deformation along their thickness. Microhardness testing was done using Vickers microhardness testing machine. Microstructure characterization of bulk aluminium and chips (as strips) was done by electron back scatter diffraction (EBSD). For in-depth deformation analysis thickness of strips was reduced in steps up to 100 μm by abrasive papers. After each step of reducing thickness of strips by 100 μm, X-ray scan and microhardness testing of strips were performed. The results show that microstrain in strips was increased and crystallite size was decreased. Moreover, hardness of the strips was ~ 40-44% more than the bulk material. Higher hardness in strips may be attributed to their ultrafine microstructure resulted from high deformation observed in strips. Furthermore, variation in deformation level along the thickness of sheets was also observed.

Keywords: Large strain machining; Severe plastic deformation; Microhardness; Microstrain; Crystallite size

1. Introduction

Enhancement in physical and mechanical properties through microstructural refinement of the materials can be achieved by inducing large plastic strain in materials. Nanocrystalline and Ultra-fine grained (UFG) materials are more wear resistant and harder as compared to their coarse grained counterparts [1]. Better properties of these materials have increased interest in production of these materials across the globe. UFG bulk materials can be produced by processing the materials by severe plastic deformation (SPD) processes. Different processes such as equal channel angular extrusion (ECAE), rolling, high pressure torsional (HPT) straining and drawing use severe plastic deformation to impose heavy deformation in materials, which further leads to their microstructural refinement [2]. These processes can be used to produce nanostructured materials from alloys of low to moderate strength. However, these processes require multiple stages for heavy deformation, making them costly for large scale production purposes. As an alternative approach, SPD can also be induced by machining-based deformation processes to produce nanostructured materials. The large deformation in chips produced during machining leads to development of ultra-fine and nanocrystalline grained microstructure in them [3]. This process can be used for producing nanostructured bulk materials from high strength materials such as Ti and Ni based alloys in a cost effective way [3].

Swaminathan et al. [4] showed microstructural phenomena in different alloys and metals associated with large strain deformation. The experimental results implied the effects of high strains, material transformation and temperature on microstructure [4]. Deformation parameters on the machined surface can be varied systematically and over a wide range. Moreover, these parameters decide the resulting texture and
microstructure in the machined surface/subsurface [5]. Shankar et al. [6] introduced different strain values in the chip by varying tool rake angle and concluded that chip of peak-aged 6061 (T6 condition) have refined microstructures and higher hardness than Al 6061 T6 subjected to equal channel angular pressing. Thermal stability of the chips with different levels of strain was also analyzed. In another investigation, Shankar et al. [7] found that the microstructure of Al 6061-T6 (Peak aged) got refined to sub-100 nm grains due to large strain machining. They concluded that there appears to be a weak but statistically significant effect of the magnitude of shear strain on the hardness of the chips [7].

Material flow and deformation tensor fields in large strain extrusion machining (LSEM) were characterized using particle image velocimetry [8]. The strain distributions in strips with shear strain < 1 were strikingly different from those of produced using free machining and shear strain > 1. Moreover, a greater depth of the workpiece was deformed to large strains for shear strain < 1. Furthermore, the strain at the surface was much larger [8]. Large shear strain along the deformation zone imposed in the chips was suggested to be the reason for grain refinement. Tribological behavior of nanocrystalline oxygen-free high conductivity (OFHC) copper and commercially pure titanium produced by LSEM was compared with the coarse grained counterparts [9]. Microstructure refinement showed no influence on friction for the materials studied under given conditions. Wear rates of nanostructured copper and titanium were lower than that of the microstructured materials [9]. The lowest wear volume was obtained when the sliding took place in the direction perpendicular to that of grain orientation. The highest wear resistance was observed for nanostructured copper material with an elongated grain structure in the extrusion direction [10]. Iglesias et al. [1, 9-10] combined large strain machining with extrusion to produce nanostrucutred foils and strips of different chip thickness ratios to induce different strain in the chips. They used different chip thickness ratios to induce different strain in chips.

Among commercial aluminium alloys, the age hardenable 6061 is widely used because it exhibits a good combination of formability, strength, corrosion resistance and weldability. Aim of current investigation is to create nanostructured strips of this alloy by large strain machining, which is a machining based deformation process. Moreover, effect of rake angle on deformation level in strips (chips) produced by large strain machining (LSM) is studied. Furthermore, in-depth analysis of the chip is done to see the variation in deformation along thickness of chip. To the best of our knowledge it is first ever attempt to do the deformation analysis of strips produced by machining using X-ray diffraction.

2. Experimental Procedure

A lathe was used as machining platform for this study. A disk of 55 mm diameter and 10 mm thickness of aluminum 6061 was used as a test material. Material was removed by plane strain machining by the radial motion of a tool into the disk at a constant feed rate of 0.44 mm/rev while the workpiece was rotating with a peripheral speed of 0.19 m/s. Since transformation of phase at high temperatures can be expected at higher cutting speed of 1-2 m/s, therefore the workpiece speed was kept low. Figure 1 shows the schematic of chip formation in plane strain machining (2-D machining) and related geometric parameters. The machining experiments were carried out with an indigenously-made LSM setup of HSS in dry environment. Rake angle was varied as +5° and 0° to impose different strain in the strips. For further reference in text, the chips produced by tool of rake angle +5° and 0° are designated as strip 1 and strip 2 respectively. Microstructure characterization of bulk aluminum and chip as strips was done by electron back scatter diffraction (EBSD). Metallographic preparation was done by polishing with abrasive papers of 200, 600, 1000, 1500, 2000 grit size followed by diamond polishing. For EBSD analysis, grounded and polished samples were further polished using electro polishing machining. EBSD scans were taken at their centres and near edges using FEI Quanta 3D FEG (Field emission gun). TSL OIM 6.1.3 software was used to find out the grain size and distribution from indexed Kikuchi patterns. The deformation behaviour of strips produced by large strain machining was investigated using X-ray diffraction (XRD). XRD analysis was performed using Panalytical X-Pert Pro diffractometer (θ = 2θ) equipped with Cu-Kα radiation (λ = 1.5418 Å). Microhardness testing was done at 100 gm load for 10 seconds using Vickers microhardness testing machine. For in-depth deformation analysis thickness of strips was reduced by 100 μm in steps using abrasive papers. Grinding of the strips was started from the surface coming in contact with the rake face of tool. In each step after reducing thickness of strips by 100 μm, X-ray scan and microhardness testing of strips were performed.

![Figure 1: Schematic of chip formation in plane strain machining showing different parameters](image)

It is well known that in X-ray diffractogram, broadening of the line is influenced by the internal strains and crystallite size. In order to obtain internal strain, Williamson-Hall method and for calculating crystallite size, Scherrer method was used [11]. In the Williamson-Hall method, the analysis includes two steps:

First step: The integral breadth of the peak was considered as width (βhkl). Integral breadth is the ratio of the area under the peak to the height of the peak. For finding out the instrumental broadening (βinh), polycrystalline silicon standard was used [12]. Then peak breadths due to sample (size + strain), B was calculated according to Gaussian profile:

\[ B = \frac{1}{2} \sqrt{\beta^2 - \beta_{inh}^2} \]
Second step: The internal strain was obtained using Williamson-Hall equation (2). Internal strain was the slope of a line fitted between points of plot between $B \cos\theta$ and $\sin\theta$

$$B \cos\theta = \left(\frac{k\lambda}{D}\right) + 2\varepsilon\sin\theta$$

(2)

where $D$ is the coherent scattering length (crystalline size); $K$ is a constant whose value is approximately 0.9; $B$ the integral width of the sample (in rad) calculated in the first step and $\varepsilon$ is the inhomogeneous internal strain (in %).

Scherrer formula (Eq. 3) was used to calculate the crystallite size

$$D = \frac{k\lambda}{B \cos\theta}$$

(3)

In this case, the peak width (in rad) was determined as the full width at half-maximum (FWHM) by a Gaussian fitting.

When a material is deformed the Bragg reflections detected with XRD can be affected in two ways. First the peaks will shift in the presence of mean strains. Second, the breadth and shape of the peaks can change for two reasons: (i) the size of diffracting elements (e.g. the grain size) changes; and (ii) changes in inhomogeneous strain (micro-strain). A possible source of inhomogeneous strain includes lattice dislocations, grain boundary defects and intracrystalline gradient such as high-dislocation-density walls surrounding low-dislocation-density cell interiors [13]. The six most intensive reflection peaks of the samples were used in the line broadening analysis. Machining imposes severe plastic deformation in the chips. Shear deformation analysis was carried out to check the level of deformation in strips produced by machining. Shear strain imposed in the chips during deformation resulted from the severe plastic deformation (secondary shear zone). This is in good agreement with the finding of Ernst [16], Merchant [17] and Sandstrom et al. [18]. The high strain produced in strips may be due to high deformation resulted from the severe plastic deformation occurred during large strain machining.

3. Results and Discussion

3.1. Shear strain analysis

Undeformed chip thickness ($t_0$) be 0.44 mm and thickness of strips ($t_c$) 1 and 2 were 0.71± 0.05 and 0.79± 0.04 mm respectively. Using these values in equation 6, a value of average chip thickness ratio ($r$) was found as 1.61 and 1.79 respectively. Average shear strain (γ) for these chip thickness ratios comes out to be 2.06 and 2.34 for the strip 1 and 2 respectively.

$$s = \frac{t_0}{5.9\sin\phi}$$

(8)

Thickness of strips was measured at different locations using Tool maker’s microscope and Vernier calliper. Average thickness of the strips was considered for the analysis.

3.2 Deformation level analysis

There is a highly localized deformation in shear zone connecting the tool tip and the workpiece surface (primary deformation zone) during the chip formation in machining, and along the interface between the chip and the tool rake face (secondary shear zone). This is in good agreement with the finding of Ernst [16], Merchant [17] and Sandstrom et al. [18]. The high strain produced in strips may be due to high deformation resulted from the severe plastic deformation occurred during large strain machining.

Fig. 2 shows the XRD patterns for the bulk aluminium and chips as strip 1 and 2. All Bragg peaks of the XRD patterns

| Sample | $2\theta$ (Deg) | Height [cts] | FWHM [2θ] | Area [cts*2θ] | Microstrain | Crystallite size (nm) |
|--------|----------------|-------------|------------|---------------|-------------|-----------------------|
| Bulk Aluminum | 38.51335 | 831799 | 0.0926 | 104036.5 |
| Strip 1 (After grinding of 100 micron thickness) | 65.1 | 61 | 0.21 | 13.77 |
| Strip 1 (After grinding of 100 micron thickness) | 78.39 | 1007 | 0.152 | 225.5 |
| Strip 1 (After grinding of 100 micron thickness) | 82.3939 | 41085 | 0.1518 | 9146.08 |
| Strip 1 (After grinding of 100 micron thickness) | 98.9802 | 7353 | 0.219 | 2310.52 |
| Strip 1 (After grinding of 100 micron thickness) | 99.031 | 116 | 0.54 | 66.22 |
| Strip 2 (After grinding of 100 micron) | 38.4506 | 12200 | 0.174 | 2716.49 |
| Strip 2 (After grinding of 100 micron) | 44.685 | 4751 | 0.201 | 1284.54 |
| Strip 2 (After grinding of 100 micron) | 65.048 | 2557 | 0.264 | 883.31 |
| Strip 2 (After grinding of 100 micron) | 78.18 | 2431 | 0.313 | 1044.48 |
| Strip 2 (After grinding of 100 micron) | 82.389 | 415 | 0.28 | 179.26 |
| Strip 2 (After grinding of 100 micron) | 99.031 | 116 | 0.54 | 66.22 |
| Strip 2 (After grinding of 100 micron) | 100.001 | 172 | 0.53 | 119.5 |
showed only the same reflections, indicating that there are no phase changes in strips during machining. In strips, the diffraction peaks became broader and their relative intensity decreases. For example, the integral breadth of the peak at 44.75° of bulk aluminium is 0.00228 rad and for the strips 1 and 2 is 0.00458 and 0.00495 rad respectively. It is well known that the broadening of the peaks can be caused by both an increase in lattice strain and reduction in crystallite size. The broadening due to strain is caused by the non-uniform displacements of the atoms with respect to their reference-lattice positions [19]. It can be observed that the strips 1 and 2 display a greater broadening than the bulk aluminium implying that in these strips size and strain changes are significant and cannot be neglected. Table 1 shows the value of FWHM of the peaks at specific 2θ angle, crystallite size and microstrain for bulk aluminum, strip 1 and 2 at different distances from surfaces.

Increase in breadth in strips is attributed to the larger grains deforming plastically, whereas smaller grains remain primarily elastic leading to inhomogeneous deformation in the strips. Figs. 3 and 4 show the microstrain and crystallite size in strips and bulk aluminium. It is observed that microstrain in strips (Fig. 3) has increased and crystallite size was decreased (Fig. 4). This may be explained through a decrease in the coherent scattering volumes due to increase in dislocation density, stacking faults, twins and related defects during deformation [20]. It has also been found that microstrain in strip 2 was more in comparison to bulk aluminium and strip 1. This can be due to high shear deformation in the strip 2. Moreover, variation in microstrain along the thickness of strips was observed. From Fig. 3 it can be noticed that maximum internal strain is at a distance of 200-300 μm from the surface. Microstrains in strips firstly increase up till depth of 200-300 μm and then it starts decreasing. This shows that deformation in strips is not uniform. Possible sources of inhomogeneous strain include lattice dislocations, grain-boundary defects and intracrystalline gradients such as high-dislocation-density walls surrounding low-dislocation-density cell interiors [13]. Inhomogeneous strain in strips may mainly be due to increase in dislocation density.

3.3 Microstructure characterization and hardness measurement

Microstructure characterization was carried out to check the effect of shear deformation on grain structure of strips. EBSD map, grain size distribution and misorientation plot for bulk aluminium, strip 1 and 2 are shown in Fig. 5. Average grain size of the bulk aluminium is found to be ~ 257 μm. It can be observed from the grain size distribution in strips that grain size in strip 2 is considerably refined with the presence of mostly ultra-fine grains (< 1 μm). Average grain size in strip 2 was found to be ~ 2.3 μm. EBSD maps of the chip particulates shows several discrete crystallographic regions, however, some of the region in EBSD map is black indicating either presence of high dislocation density is source of incoherency or it can be due to difficult to detect small crystalline regions. The misorientation distribution in strips illustrates the presence of large fraction of low angle grain boundaries (LAGB) relative to that for high angle grain boundaries in bulk aluminium.
### Figure 5: EBSD map, grain misorientation distribution and grain size distribution of bulk aluminum 6061 and chips produced with tool of rake angle +5° (strip 1) and 0° (strip 2)

**EBSD Map**

**Grain size distribution and Misorientation plot**

- **Bulk Aluminum**
- **Strip 1**
- **Strip 2**

| Grain Size Diameter (μm) | Area Fraction |
|--------------------------|---------------|
| 15                       | 0.00          |
| 65                       | 0.05          |
| 110                      | 0.10          |
| 160                      | 0.15          |
| 210                      | 0.20          |
| 260                      | 0.25          |
| 310                      | 0.30          |
| 355                      | 0.35          |
| 400                      | 0.40          |
| 470                      | 0.45          |

| Misorientation Angle (Degree) | Area Fraction |
|-------------------------------|---------------|
| 0                             | 0.00          |
| 1                             | 0.05          |
| 2                             | 0.10          |
| 3                             | 0.15          |
| 4                             | 0.20          |
| 5                             | 0.25          |
| 6                             | 0.30          |
| 7                             | 0.35          |
| 8                             | 0.40          |
| 9                             | 0.45          |
| 10                            | 0.50          |
| 11                            | 0.55          |
| 12                            | 0.60          |

| Grain Size Diameter (μm) | Area Fraction |
|--------------------------|---------------|
| 3                         | 0.00          |
| 7                         | 0.10          |
| 10                        | 0.20          |
| 13                        | 0.30          |
| 16                        | 0.40          |
| 22                        | 0.50          |
| 26                        | 0.60          |
| 28                        | 0.70          |
| 32                        | 0.80          |
| 35                        | 0.90          |
| 42                        | 1.00          |

| Misorientation Angle (Degree) | Area Fraction |
|-------------------------------|---------------|
| 3                             | 0.00          |
| 7                             | 0.05          |
| 13                            | 0.10          |
| 16                            | 0.15          |
| 19                            | 0.20          |
| 22                            | 0.25          |
| 26                            | 0.30          |
| 28                            | 0.35          |
| 32                            | 0.40          |
| 35                            | 0.45          |
| 42                            | 0.50          |
| 44                            | 0.55          |
| 47                            | 0.60          |

| Grain Size Diameter (μm) | Area Fraction |
|--------------------------|---------------|
| 15                        | 0.00          |
| 65                        | 0.05          |
| 110                       | 0.10          |
| 160                       | 0.15          |
| 210                       | 0.20          |
| 260                       | 0.25          |
| 310                       | 0.30          |
| 355                       | 0.35          |
| 400                       | 0.40          |
| 470                       | 0.45          |

| Misorientation Angle (Degree) | Area Fraction |
|-------------------------------|---------------|
| 0                             | 0.00          |
| 1                             | 0.05          |
| 2                             | 0.10          |
| 3                             | 0.15          |
| 4                             | 0.20          |
| 5                             | 0.25          |
| 6                             | 0.30          |
| 7                             | 0.35          |
| 8                             | 0.40          |
| 9                             | 0.45          |
| 10                            | 0.50          |
| 11                            | 0.55          |
| 12                            | 0.60          |
It implies that grains in strips are aligned in a direction as compared to highly misoriented grains in bulk alloy. Comparison of grain size distribution at different locations in strips revealed high variation in grain size with a grain as small as ~100 nm and grain as large as ~4 μm. Results show that strip 2 has a relatively refiner grain structure in comparison with strip 1. Among the investigated cases, the strip 2 has the most refined microstructure. Large number of pits or voids mostly submicron in size were observed throughout the machined strips. Significant number of voids in machine chip might be due to the severe plastic deformation (SPD) occurred in chips during machining. Moreover, large shear strain produced in the strips can be the reason for producing high densities of crystal lattice defects such as dislocations, which can further lead to grain refinement.

Grain refinement in chip particulates was further supported by high hardness value of strips in comparison to the bulk aluminum. Vickers hardness of the strips was measured as $114 \pm 7$ HV and $120 \pm 8$ HV respectively, indicating around 40% and 44% increase over the hardness of bulk aluminum ($84 \pm 3$ HV). The high hardness of strips produced using large strain machining may be attributed to their fine grain microstructure resulting from the severe plastic deformation during machining. Moreover, the hardness variation along the thickness of the strips is not significant. It is worthwhile to mention that such nanostructured strips produced by machining based deformation processes can be further be used for manufacturing of micro components [21].

4. Conclusions

Present work is focused on in-depth deformation analysis in chips produced by machining. Moreover, deformation induced microstructure found to be influenced by level of strain resulted from different tool rake angle.

- Nanostructured aluminum strips in the form of chips were produced using a large strain machining at different rake angles.
- From XRD line broadening analysis, it was observed that microstrain in strips was increased, while crystallite size was decreased.
- The rake angle was found to influence the Microstrain and grain size of the strips. Whereas the effect on crystallite size was insignificant. As the rake angle was changed from +5° to 0°, the grain size of the strips was found to refine.
- Vickers hardness of the fabricated aluminum strips was around 40- 44% higher than that of bulk aluminum, which may be attributed to finer microstructure of the former.

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