Relating microtexture and dynamic micro hardness in an extruded AA8090 alloy and AA8090-8 vol% SiCp composite

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Abstract

The present study involves combined measurements of microtexture and dynamic ultra micro hardness (DUH) in hot extruded AA8090 aluminum alloy and its composite reinforced with 8 vol% SiCp. Both the materials show strong crystallographic fiber textures—\(h_{111}\) and \(h_{001}\). The dynamic micro hardness shows a clear pattern of difference between these two fiber textures, \((111)\) oriented grains being harder and stiffer. The difference in \(q/d\) between the fibers, where \(q\) and \(d\) are the average cell misorientation and cell size, respectively, was marginal in the alloy and thus cannot explain the observed hardness difference. The hardness difference can be explained from the difference in Taylor factors between the respective fibers. Elastic stiffness values estimated from microtexture and DUH follow a similar trend qualitatively.

Keywords: Texture; EBSD; Dynamic microhardness; Al–Li alloy; Particle reinforced composite

1. Introduction

The constant need for higher fuel efficiency in aerospace and automobile industries was prime mover for the development of low-density Al–Li alloys. An addition of 1% Li can reduce the density by about 3% and at the same time leads to increase in strength and stiffness [1]. To increase the strength and stiffness further, it is quite logical to think of reinforcing Al–Li alloys with ceramic particles. Such composites are often formed through hot extrusion. Extrusion of Al alloys and composites typically produces a structure with marked directionality [2]. Original grains are elongated along the extrusion direction together with the reinforcing particles. Though mechanical properties are often considered to be anisotropic and related to the bulk crystallographic texture [3–5], little is known on the possible linking between micro-mechanical response and the local orientation.

Indentation hardness technique has been in use for many years to evaluate the mechanical properties of materials [6–9]. Dynamic Ultra Low Load Micro Hardness (DUH) indentation offers considerable advantage over more conventional static indentation technique. DUH primarily consists of a controlled load \((P)\) applied through a diamond tip that is in contact with a smooth surface. The penetration depth is continuously recorded as a function of load. In this technique, dynamic microhardness values are obtained from the in situ depth measurements during indentation. In situ depth measurement permits use of very low loads and yields accurate hardness and elastic response values. In other words, DUH can be used effectively to bring out micro-mechanical behavior in a material system.

Emergence of automated indexing of backscattered Kikuchi diffraction led to the realization of orientation imaging microscopy (OIM) [10–12]. In this technique, fast pattern acquisition coupled with automated indexing and beam movements provide a complete mesoscopic microstructure, where different features are distinguished based on their microtextural differences. The microtextural information can also be used to estimate the trends in micro-mechanical properties—plastic and elastic [13]. The broad objective of the present study was to explore the existence, if any, of linkages between OIM estimated micro-mechanical properties and the properties evaluated by DUH measurements.

2. Experimental procedure

An 8090 Al–Li alloy and its composite reinforced with 8 vol% SiC particles have been used in this study.
Table 1
Composition of the alloy

| Li | Cu | Mg | Zr | Fe | Si | Al |
|----|----|----|----|----|----|----|
| 2.14 | 2.0 | 0.88 | 0.12 | 0.1 | 0.03 | Bal. |

The composition of the alloy is given in Table 1. SiC particles of average size 40 μm was used as reinforcement. The composite used in this study was fabricated by stir casting technique. Cast billets were hot extruded in a CBJ 250 T extrusion press. The temperature range in which an alloy or composite can be extruded is determined mainly by the onset of undesirable processes such as surface cracking, chevron cracking, incipient melting or significant recrystallization. The optimum extrusion temperatures for the alloy and its composite used in this study were found to be 475 and 530 °C, respectively. An extrusion ratio of 22:1 was used.

The extruded rod was cut along the extrusion direction by a slow speed diamond wheel cutter. Samples were polished up to 1200 grit emery paper and fine polished to mirror finish with diamond paste. These were then electro-polished at −20 °C using an electrolyte of methanol: perchloric acid (80:20) and 11 V dc. Areas were marked by lacquer on these samples and OIM scans were made over an area of at least 1 × 2 mm² in each sample on the longitudinal section (having extrusion and transverse/normal direction). Microtexture measurements were carried out using a TSL OIM system on a FEI Quanta 200 HV SEM.

The areas used for OIM measurements, or rather grains with distinct crystallographic fibers identified from OIM analysis, were then subjected to DUH measurements. The DUH measurements were carried out on a Simadzu Dynamic Ultra Low Load Micro Hardness tester (DUH-202). The locations for measurements were fixed with the help of two micrometers having an accuracy of 0.01 mm. In this technique, dynamic microhardness values are obtained from the in situ depth measurements during indentation. In situ depth measurement permits use of very low loads and yields accurate hardness and elastic response values. A Vickers indenter with an included angle of 136° between the faces was used for indentation. A load of 2 gm was applied for duration of 5 s. Typically indentations were made at an interval of 10 μm along the extrusion direction. At least 300 μm length (and measurements were repeated at several grains) was covered in each set of measurements. The values reported in the present study represent average of at least three sets of measurements.

3. Results

3.1. Microtexture

Both the materials show two predominant fiber orientations, (111) and (001), as shown in Fig. 1. The extents of texturing are not too different—maximum ODF (orientation distribution function) intensity (being a relative index of anisotropy or crystallographic texturing) of the alloy and the composite being 16.7 and 15.5, respectively. The difference in texture is mainly through relative presence of (111) and (001) fibers. To bring this aspect more clearly, the alignment (with respect to ideal fiber orientation) and the relative ratio of (111)(uvw) and (001)(uvw) fibers are plotted—see Fig. 2. In the figure, x-axis is taken as alignment—calculated from (uvw)ideal and (uvw)sample for each discrete data point. The y-axis, on the other hand, contains the ratio of (111)(uvw) and (001)(uvw) fibers, the ratio being estimated from the number fraction of discrete data points falling within specific alignment. As shown in Fig. 2, the ratio of these two fibers is similar in the alloy and composite for alignments of up to 5°. For larger angles of alignment, however, the alloy clearly exhibits a stronger (111) fiber texture, while the relative fiber strength for the composite remains unaffected.

Fig. 3 shows the OIM images of the composite. As shown in the figure, the SiC particles were clearly visible and so were the long grains of the respective fiber families. Fig. 3(a) shows the image of detector signal. Fig. 3(b) and (c) are the ‘zoomed’ up images (taken from the area marked in Fig. 3(a)) showing the respective fibers (see Fig. 3(b)) and the Taylor factor maps (see Fig. 3(c)). Similar measurements were obtained from the alloy as well.

3.2. DUH measurements

The DUH measurements were taken from the well-defined grains/bands of respective fibers, as shown in Fig. 3. Fig. 4
shows the dynamic Vickers microhardness (DHV) values along \(h_{111}\) and \(h_{001}\) oriented grains (within 10° of ideal fiber orientation) in the unreinforced alloy and the composite. It is to be noted that several of such measurements were made to arrive at statistically consistent trends. The DUH data was interpreted for two information—the hardness and the elastic modulus values. The latter was calculated by fitting the load-displacement data to a model [7]. These values, along with their standard deviations, were estimated for grains of the respective fibers, \(h_{111}\) and \(h_{001}\), and are listed in Table 2. In order to compare these two parameters (hardness and modulus) several parameters were estimated from the microtexture measurements and these are also included in Table 2.

### 3.3. Combining microtexture and DUH data

The OIM data was also filtered or partitioned for \(h_{111}\) and \(h_{001}\) oriented grains. Grains within the same fiber (texture) family were identified on the basis of a maximum deviation or misorientation of 10°. The average misorientation, \(\theta\), between the neighboring grains and the sub-grain size, \(d\), (2° misorientation criterion was applied to define sub-grain) were calculated from the respective partitions.

The stored energy of cold work is expected to scale with \(\theta/d\) [14]. In the present study, the \(\theta\) and \(d\) values were estimated from the OIM scans and are listed for the respective fibers in Table 2. The difference in \(\theta/d\) values may be stipulated to tally with the hardness differences provided the main cause of such hardness difference is from the substructure or stored energy of cold work. In the case of the composite, a difference of about 10% for both \(\theta/d\) and hardness does exist between \(h_{111}\) and \(h_{001}\) oriented grains, see Table 2. In the case of the alloy, on the other hand, the difference in \(\theta/d\) hardly exists between the fiber textures, while a significant difference (of about 18%) in hardness was observed. Therefore, the difference in \(\theta/d\) cannot be related to the observed hardness difference and the only way to explain this difference is from Taylor factor. Taylor factor and elastic modulus values were calculated for the respective fibers or grains using simple compression as the strain matrix. Arguably the indentation is of complex triaxial strain, but a simplified approach of simple compression strain path was taken. The OIM software calculates [13] the Taylor factor using appropriate slip system and the full constraint Taylor model, while stiffness tensor is calculated using Voigt and Reuss method [15] taking single crystal elastic constants and local orientation as inputs. Voigt and Reuss stiffness tensors can be averaged to calculate an approximate polycrystal average stiffness tensors—Bishop-Hill average [13]. The values of the Taylor factor and the elastic modulus estimated from OIM are given in Table 2. Naturally, the values of Taylor factors were same for the alloy and the composite, as the role/existence of second phase particles cannot be incorporated in the Taylor type model used in OIM analysis. It is interesting to note that elastic modulus estimated from microtexture and DUH follow a similar trend qualitatively (not in an exact quantitative way though), \(h_{111}\) oriented grains being stiffer compared to \(h_{001}\) oriented grains.

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**Fig. 3. Typical OIM scans from the composite.** (a) Image from the detector signal clearly identifying the SiC particles. ‘Zoomed’ up images (from the area marked in Fig. 3(a)) of the (b) respective fibers and (c) Taylor factors. In (b) the grain boundaries above 20° misorientation are marked. In (c) black regions indicates Taylor factors of 2.27–2.55 (corresponding to \(h_{001}\) fiber), while Taylor factors of \(h_{111}\) fibers (white regions in figure (c)) were between 3.39 and 3.67.
4. Discussion

Control of bulk mechanical properties, such as formability, through control of bulk crystallographic texture is not too uncommon [16]. In the same token, one may expect a relationship between microtexture and micro or mesoscopic mechanical properties. The objective of the present study was to explore the validity of such a relationship. The alloy and the composite have gone through severe deformation due to the process of extrusion, hence the process is expected to cause significant texturing and highly elongated bands/grains with fiber textures (see Fig. 1). The two fibers, in both alloy and composite, show nearly similar texturing or maximum ODF intensity. Typically the entire band stayed within an orientation spread of 10–15°.

The textural details, as shown in Figs. 1 and 2, especially the alignment with respect to ideal [111] and [001] fiber texture pose some interesting questions. But such questions are beyond the scope of the present study. It also needs to be pointed out that other than difference in deformation behavior (and expected differences in deformation texture developments) between the alloy and the composite, the optimized deformation conditions (as used in the present study) were also different between the two, making a direct comparison difficult.

It is also necessary to highlight, at this point, the limitations of the two characterization techniques (OIM and DUH) used in the present study. There are two possible biases for DUH measurements. First, in case of composites, effect of proximity to SiC particles on DUH measurements, especially from down below, can never be fully ruled out. This difficulty is expected to be relatively less in the alloy, where second phase particles are typically sub-micron in size. The other issue for DUH data is the data interpretation. For minor load/depth, relaxation of the indenter tip may add to the inaccuracy/scatter, especially in the fitting approach [7] used in the estimation of elastic modulus. The enhancement of modulus of a composite as measured by indentation technique depends largely on presence of subsurface reinforcement particles beneath the indentation and proximity of particles. In case of very low loads (as in the present study) hard particles at the subsurface may not be encountered. In the composite studied in the present study the maximum depth of indentation is 0.68 μm and the diagonal length of indentation is 5 μm whereas the average spacing between SiC particles is 60 μm. Hence, the possibility of indenter encountering a SiC particle beneath the indentation is very remote. Further, the grains of fiber texture are almost free of SiC particles (as can be seen from Fig. 3(b)). The lower modulus values of the composite compared to the alloy (though the difference falls well within the experimental scatter) may be attributed to these two factors. Moreover, as discussed above for minor load/depth (as in the present study), the relaxation of the indenter tip may add to the inaccuracy.

Table 2
Summary of microtexture (OIM) and dynamic ultra micro hardness (DUH) measurements

| Parameters                          | Alloy (111)grains | Alloy (001)grains | Composite (111)grains | Composite (001)grains |
|-------------------------------------|------------------|------------------|----------------------|----------------------|
| Average misorientation among neighboring subgrains (°) | 3.2 (0.5) | 2.5 (0.6) | 1.67 (0.4) | 1.47 (0.5) |
| Average cell/grain size, d (μm)     | 10.8 (0.8) | 8.4 (1.6) | 5.5 (0.2) | 5.3 (0.2) |
| \(\theta/d\)                        | 0.296         | 0.298           | 0.304                | 0.277                |
| Taylor factor (M)                   | 3.53 (0.2) | 2.41 (0.2) | 3.53 (0.2) | 2.41 (0.2) |
| Elastic modulus (GPa)               | 127 (9)      | 87 (7)          | 127 (9)              | 87 (7)               |
| DUH Hardness (DHV)                  | 132 (10)     | 112 (7)         | 159 (10)             | 145 (8)              |
| Elastic modulus (GPa)               | 101 (12)     | 94 (13)         | 96 (8)               | 84 (8)               |

Average values as well as the standard deviations (in bracket) are given. Taylor factors and the elastic modulus values estimated from OIM were same for respective fibers of the alloy and the composite.
or scatter, especially in the fitting approach used here to determine the modulus. This approach determines the modulus based on the contact stiffness [7] which is affected significantly by the relaxation of the indenter tip.

The estimation of the \( \theta/d \) from microtexture data was direct and the low values of standard deviation provide an indication of relatively insignificant scatter. Questions, however, can be raised on the indirectly estimated Taylor factor and elastic modulus values. Firstly, the strain path of the indentation hardness was simplified as simple compression, as an exact knowledge of the actual triaxial strain path does not exist. Secondly, both the Taylor factor and the elastic modulus were estimated from continuum based models. The ‘Taylor factor’ (\( M \)) is frequently used to express a flow stress, \( \sigma \), measured in a polycrystal in terms of the critical resolved shear stress (CRSS), \( \tau \), in the constituent single crystals [17]. It is an average orientation factor, which depends on the texture of the material and on the crystallographic nature of the slip systems [13,17]. The Taylor factor has been used to explain relative differences in stored energy of deformation between different orientations, both macroscopically [18–20] and microscopically [16,21], or even to explain the structural developments during deformation [16,21]. This works quite well for orientation with extreme difference in Taylor factors, such as Cube (100)(001) and S (123)(634) in fcc aluminum [14,21], but may not be so successful for orientation with relatively minor differences [21]. Fortunately, the families of fiber textures used in the present study are with large differences in Taylor factors, as shown in Table 2. Reported difference in nano-hardness [22] is about 60% between (111) and (100) single crystal thin films of pure aluminum. As shown in Table 2, this is close to the difference in Taylor factor between the two orientations. The estimated hardness differences between the (111) and (001) grains in the alloy and the composite are 18 and 10%, respectively, which is significantly lower than the difference in Taylor factors and the reported values in single crystal pure aluminum. The extruded alloy and the composite are commercial systems with substructure and presence of second phase particles and precipitates (especially sub-micron particles). These two factors are expected to enhance the absolute hardness of the material, including the hardness of the individual fibers. An increase in absolute hardness, in turn, is expected to reduce the hardness difference (in percentage) between the fibers. Use of elastic modulus in OIM mapping is relatively recent. Though this can be used to map elastic anisotropy, the use of this technique to relate to experimental elastic anisotropy does not exist.

The relatively minor difference in hardness between the grains with \((111)\) and \((001)\) texture of the composite (as compared to the alloy) can possibly be explained from the ‘equalizing’ effects provided by the presence of SiC particles. It is to be noted that a classical Taylor model does not take the presence of second phase into account. For the alloy, however, the large difference in hardness can only be explained from the estimated differences in Taylor factors, the \( \theta/d \) difference being insignificant. Although, there is no quantitative agreement between the elastic modulus values obtained through DUH and OIM, an overall agreement of trends could still be observed in a qualitative manner. In other words, in spite of the limitations of the DUH and microtexture data and the fact that the material is a commercial system, an approximate linkage between orientation dependent parameters (Taylor factor and elastic modulus) and related properties estimated by DUH could be established.

5. Conclusions

1. Hot extruded 8090AI alloy and 8090Al-8 vol% SiC\(_p\) composite contain grains of predominantly two fiber families, namely (111) and (001).
2. For both the alloy and the composite, the DUH estimated hardness and elastic modulus were higher in (111) fiber compared to (001).
3. A difference of about 50% in Taylor factor was estimated between (111) and (001) oriented grains. This difference is weakly reflected on the difference in hardness values of these fibers, which is 18 and 10% in the alloy and the composite, respectively.
4. Qualitatively (and not in an exact quantitative way) the elastic modulus estimated by DUH and OIM for the respective fibers also follow a similar trend.
5. An approximate linkage could be worked out between microscopic hardness and elastic modulus values, as obtained through DUH, and orientation dependent values of Taylor factor and elastic modulus, as obtained through OIM.

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