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Procedia Materials Science 5 (2014) 379 – 386
Procedia Materials Science
www.elsevier.com/locate/procedia

International Conference on Advances in Manufacturing and Materials Engineering, AMME 2014

Evaluating the mechanical properties of HPT processed aluminium alloys using automated ball-indentation technique

Deepak C. Patil[a,*], Mousumi Das[b], Goutam Das[b], S. A. Kori[c], K. Venkateswarlu[d],

[a]*KLE Dr. M.S. Sheshgiri College of Engineering and Technology, Udyamabag, Belgaum-590008, Karnataka, India*
[b]*CSIR-National Metallurgical Laboratory, Jamshedpur-831007, India*
[c]*Basaveshwar Engineering College, Bagalkot-587102, Karnataka, India*
[d]*CSIR- National Aerospace Laboratories, Bangalore-560017, India*

Abstract

Al-Cu and Al-Zn-Mg alloys exhibit excellent physical and mechanical properties, and are widely used for aerospace applications. However, these properties may be further improved through high-pressure torsion (HPT) processing. HPT processing was carried out on Al-2014 alloy and Al-7475 alloy to investigate the improvement in mechanical properties. Disks of coarse-grained Al-2014 alloy and Al-7475 alloy were processed up to two turns of HPT, and the mechanical properties were evaluated using automated ball indentation (ABI) technique. HPT processing was carried out at atmospheric temperature, under pressure of 6 GPa. The results show that, for Al-2014 alloy the ultimate tensile strength at the edge of HPT disk increased by a factor of ~ 4, after 2 turns of HPT processing. Since, ABI technique provides the capability of obtaining extensive mechanical data using only a very small sample; it is an ideal procedure for evaluating the mechanical properties of samples processed by HPT.

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Keywords: High pressure torsion; Automated ball indentation; Mechanical properties; Al-Cu alloy; Al-Zn-Mg alloy

1. Introduction

Severe plastic deformation (SPD) has been developed in the previous two decades as a "top-down" approach for producing bulk ultrafine-grained or nanostructured materials with enhanced physical, mechanical and multi-

* Corresponding author. Tel.: +91-94-48-446146; fax: +91-0831- 2441644.
E-mail address: todeepakpatil@rediffmail.com

2211-8128
Selection and peer-review under responsibility of Organizing Committee of AMME 2014
doi:10.1016/j.mspro.2014.07.280

functional characteristics. In order to transform a coarse grained metal or alloy in to a material with ultrafine or nanoscale grains through top-down approach, it is very essential to impose very high strains in the material so as to introduce a high density of dislocations which subsequently re-arrange to form an array of grain boundaries in the microstructure. Compared to other SPD techniques, high-pressure torsion (HPT) is probably the most efficient technique for grain refinement, since extremely large strain and high pressure can be applied to specimens more conveniently compared to other SPD techniques, such as the well-known repetitive corrugation and straightening (RCS), equal channel angular pressing (ECAP) and accumulative roll bonding (ARB).

The high strains induced by SPD lead to high densities of defects, including dislocations and twins in microstructures, and substantially refine coarse grains into ultrafine (<1μm) or nanocrystalline (<100 nm) grains [Langdon, 2013]. Satisfactorily large strains can be imposed for the enhancement of mechanical properties together with the refinement of microstructure, as the dimensions of the specimens are not altered after SPD processing. Four mechanisms have been reported for the SPD induced modification of precipitate microstructure. This includes (i) the fragmentation of pre-existing precipitates due to a high strain applied by the processing, (ii) accelerated precipitation, which is observed in Aluminium alloys processed by SPD at relatively high temperatures, (iii) the dissolution of precipitates that occur during SPD processing at low/ambient temperatures, and (iv) changing the orientation of precipitates in the matrix. This leads to isotropic growth of the precipitates into a spherical morphology due to the loss of the low-energy interface between the precipitates and the Al matrix. In contrast to the conventional processes of high deformation like rolling or wire drawing, which strongly change the geometry of the sample, SPD techniques do not involve changes in the material geometry. SPD techniques today, are developing very fast and are moving from laboratory scale research in to commercial production.

Repetitive corrugation and straightening (RCS), is the SPD technique that can create ultrafine grained materials free of porosity, and also can be easily applied to large scale industrial production [Rajinikanth et al., 2008]. This involves the repetitive corrugation of a sample in 3-point bending and the subsequent straightening between two flat platens [Pandey et al., 2012]. It was noted in the early reports that the RCS process is essentially discontinuous but it may be methodically scaled-up to give continuous processing through the use of corrugated rollers in place of the traditional rollers in an industrial rolling facility and then passing the corrugated sheet between flat rollers. HPT is particularly attractive among the various routes of SPD, owing to the small grain sizes which can be achieved by this method and also as it permits a defined continues variation of strain whereas most SPD processes offer only a stepwise application of strain. The principle of HPT is that a sample, generally in the form of a thin disk, is subjected to a high pressure between two massive anvils and then processed through the application of torsional straining. In practice, the shear strain, imposed on the HPT sample is given by the relationship,

$$\gamma = \frac{2\pi N r}{h} \tag{1}$$

Where N is the number of HPT turns, r is the radial distance from the center of disk and h the disk thickness. Further, using the relationship of $\epsilon = \gamma/\sqrt{3}$, the equivalent Von Mises strain imposed on the disk, is given by,

$$\epsilon = \frac{2\pi N r}{h\sqrt{3}} \tag{2}$$

It can be very clearly seen form the Equation (1) and Equation (2) that the equivalent Von Mises strain imposed on the HPT disk increases from the center of disk towards the circumference.

Nomenclature

SPD	Severe Plastic Deformation
HPT	High Pressure Torsion
RCS	Repetitive Corrugation and Straightening
ECAP	Equal Channel Angular Pressing
ARB	Accumulative Roll Bonding

2. Methodology

In this investigation, automated ball-indentation (ABI) technique is utilized for the determination of mechanical properties of the various HPT processed samples. ABI technique is a non destructive procedure for the determination of mechanical behavior of materials, which can be utilized for the situations where *in situ* examination of mechanical behavior is necessary. ABI utilizes very small surface area of the sample, during mechanical behavior determination. This technique works on the principal based on the strain managed several indentations at a single penetration position on a polished surface by a small spherical indenter. The indenter depth is gradually increased to a maximum user specified limit with intermediate partial unloading. In these experiments, a WC ball with 1.57 mm diameter was indented on each test sample, at the center and as well at a point near the edge, using an indentation speed of 0.5 mm min^{-1}. A load cell of 2500 N and loads up to 1000 N were used in these experiments. A velocity of 0.5 mm min^{-1} was maintained during repetitive sequences of loading, unloading and reloading so as to set up multiple load-deflection curves. Using the software, the machine automatically plots curves of true stress Vs true plastic strain, from these load-deflection curves. Finally, the yield strength, the ultimate tensile strength, strain hardening coefficient, strength coefficient and Brinell hardness number (BHN), for the HPT processed samples were obtained from the true stress Vs true plastic strain curves [Venkateswarlu et al., 2006].

Fig.1 shows the indentation profiles during the application of load and once after the load is released, for a typical loading-unloading sequence employed in ABI technique. Here h_t and d_t are the total depth and the diameter during load application, and are measured for every loading cycle. Similarly h_p and d_p are the plastic indentation depth and projected diameter once the load is released. ABI technique utilizes these values of h_t , h_p along with the corresponding loads in *N*, for determining various mechanical properties of materials.

The homogeneous plastic flow portion of the true-stress (σ_t) Vs true-strain (ε_p) curve can be represented by the following familiar power law equation,

$$\sigma_t = K \epsilon_p^n \tag{3}$$

Where, n = strain-hardening coefficient and K = strength coefficient. Through the regression analysis of the above power law equation for different values of true-stress (σ_t) and true-strain (ε_p), the values of strain-hardening coefficient (n) and the strength coefficient (K) can be determined. Computer program is used to solve the following equations and to thereby determine the flow curve from the ABI technique data.

$$\epsilon_p = 0.2 \times \frac{d_p}{D} \tag{4}$$

$$\sigma_t = \frac{4P}{\pi d_p^2 \delta} \tag{5}$$

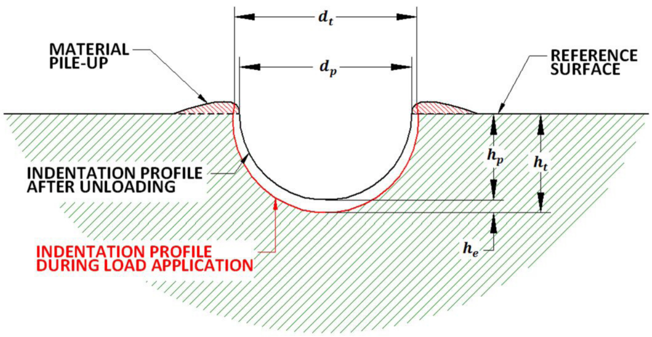


Fig. 1. Indentation profiles during the application of load and once after the load is released

Where, P = applied load and d_p= plastic indented diameter. If E_1 and E_2 are the Young's modulus of the indenter and the specimen tested respectively then the plastic indented diameter can be obtained by,

$$d_p = \sqrt[3]{2.735P \left(\frac{1}{E_1} + \frac{1}{E_2}\right) D \left(\frac{h_p^2 + 0.25d_p^2}{h_p^2 + 0.25d_p^2 - h_pD}\right)} \tag{6}$$

In the Equation (5), δ is constraint factor which can be found from the Equation (7).

$$\delta = 2.87 \times \alpha_m \tag{7}$$

The parameter α_m in the above equation depends mainly on the work hardening characteristic and strain rate sensitivity of the material being tested. The value of α_m varies from 0.9 to 1.25 for different materials and it has the value of 1.0 for low strain rate sensitive materials. The values of ultimate tensile strength σ_{uts} and the Brinnel hardness number (BHN) are given by the expressions,

$$\sigma_{uts} = K \left(n/e\right)^n \tag{8}$$

$$BHN = \frac{2P}{\pi.D\left(D - \left(D^2 - d_t^2\right)^{0.5}\right)} \tag{9}$$

Further, the data points from all the loading cycles are fit by linear regression analysis to the following relationship of Meyer [Meyer, 1908].

$$\frac{P}{d_t^2} = A \times \left(\frac{d_t}{D}\right)^{m-2} \tag{10}$$

Where m = Meyer's coefficient, which generally has a value between 2 to 2.5 and A = material parameter. Finally, the yield strength of material can be obtained using the following equation:

$$\sigma_y = \beta_m \times A \tag{11}$$

In Equation (11), parameter β_m is a constant for a given class of material. The value of β_m for each class of material is determined from the yield strength obtained using standard tensile tests. Then this β_m will be the same for a specific class of materials irrespective of heat-treatment and mechanical working.

3. Experimental material and procedures

The commercial Al-2014 alloy, processed by HPT contains in wt%, 5.0 % Cu, 0.98 % Si, 0.75 % Mn, 0.64 % Mg, 0.37 % Fe, 0.09 % Zn, 0.01 % Cr, and 0.15 % others with the remaining as aluminium. This is a high strength alloy at higher temperatures and also at ambient temperature. It shows wonderful finish ability, better workability and also good machinability. The second alloy processed by HPT was Al-7475 alloy, which is a derivative of Al-7075 alloy; containing, in wt%, 6.2 % Zn, 2.6 % Mg, 1.9 % Cu, 0.25 % Cr, 0.12 % Fe, 0.10 % Si, 0.06 % Mn and 0.15 % others with the remaining as aluminum. Al-7475 also is a high strength alloy, with appreciably higher level of fracture toughness. As both of these alloys find wide applications in many aerospace structural elements, further improvement in the mechanical properties of these alloys has always attracted sufficient research work. Circular disks of Al-2014 alloy and Al-7475 alloy were prepared with a thickness of ~ 0.83 mm and a diameter of 10 mm. These disks were processed by quasi-constrained HPT, under an imposed pressure of 6 GPa and at a rotational speed of 1 rpm. The details of HPT processing were described in earlier report [Rajinikanth et al., 2011]. Separate disks were processed through ¼, ½, ¾, 1 and 2 revolutions of HPT at ambient temperature. During processing care was taken to see that the disk is under strict confinement and no slippage occurs during HPT between the disk and the two anvils. Also proper alignment between the upper and the lower anvils was verified during processing of every sample, as any misalignment or eccentricity between the anvils would cause asymmetric processing of the samples.

Based on the geometry of the anvil and the following restrictions on lateral flow of the material, the precise characteristics of HPT processing may be divided into three separate categories [Hohenwarter et al., 2009]. In practice, however, HPT is normally conducted using a quasi-constrained facility in which the disk is positioned between depressions in the upper and lower anvils and these partial side walls constrain the sample but permit some outward flow of material in the small gap between the two anvils. In case of unconstrained HPT, the disk is placed on a flat anvil surface where it is free to flow outwards under the applied pressure and there is a monotonic thinning of the disk during processing, which contrasts with constrained HPT where the sample is held in place within a depression without any possibility of outward flow of material. The major advantage of the HPT process is that it applies maximum strain compared to all other SPD techniques, in a very simple fashion [Ligda et al., 2012]. The compressive load applied in case of HPT produces compressive stresses that are very effective in preventing the cracking of sample and the torsional straining of thin disk achieved by the rotation of anvil produces severe plastic deformation [Skrotzki et al., 2013]. The thickness of HPT sample is limited in order to ensure homogenous deformation in the axial direction throughout the volume of specimen. Following HPT, each disk was polished carefully using 1200 grit emery paper and an alumina 0.02 μm suspension. Care was taken to avoid any heating during polishing. Using ABI technique the mechanical properties of the HPT processed specimens were evaluated.

4. Results and discussions

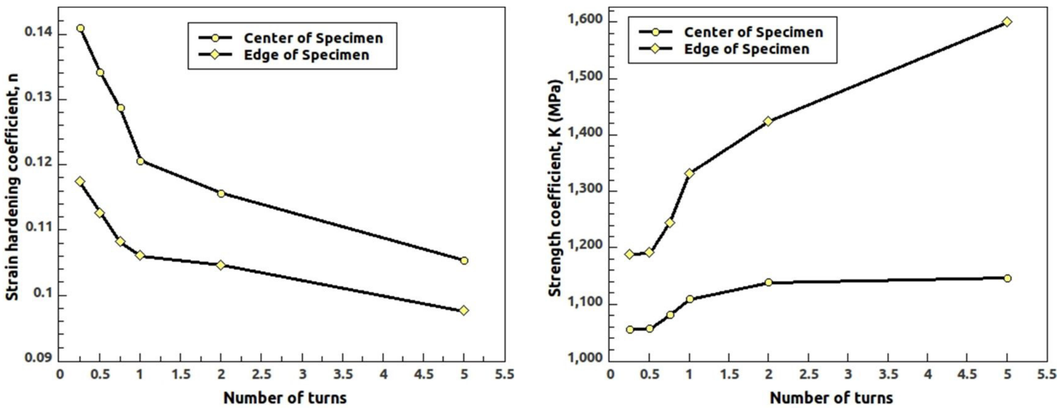
4.1. ABI technique results for HPT processed Al-2014 alloy

The ABI technique results for Al-2014 alloy, processed by various number of HPT turns are summarized in Table 1. The values of strength coefficient (K), strain hardening coefficient (n), yield strength (YS), ultimate tensile strength (UTS) and Brinell hardness number (BHN) attained by the disks are tabulated against the various number of HPT turns.

Table 1 ABI technique results of HPT processed Al-2014 alloy

No. of Turns	Position	K (MPa)	n	YS (MPa)	UTS (MPa)	Hardness (BHN)
1/4 turn	Center	1056	0.141	276	696	156
	Edge	1189	0.118	319	823	179
1/2 turn	Center	1057	0.134	282	706	159
	Edge	1192	0.113	337	833	181
3/4 turn	Center	1082	0.129	291	731	163
	Edge	1245	0.108	352	879	188
1 turn	Center	1110	0.121	311	762	169
	Edge	1333	0.106	386	945	198
2 turns	Center	1139	0.116	325	791	173
	Edge	1425	0.105	412	1014	207

The variation of strain hardening coefficient and strength coefficient for Al-2014 alloy is plotted in the Fig. 2 against the increasing number of HPT turns, whereas Fig. 3 shows the continues enhancement of yield strength and ultimate tensile strength with increasing number of HPT turns. The first important conclusion from inspection of Fig. 3 is that, as the number of HPT turns are increasing, there is continues enhancement of both the ultimate tensile strength and yield strength of Al-2014 alloy. For every disk processed by HPT the yield strength and the ultimate tensile strength are higher at the circumference of the disk compared to their values at the center, irrespective of the



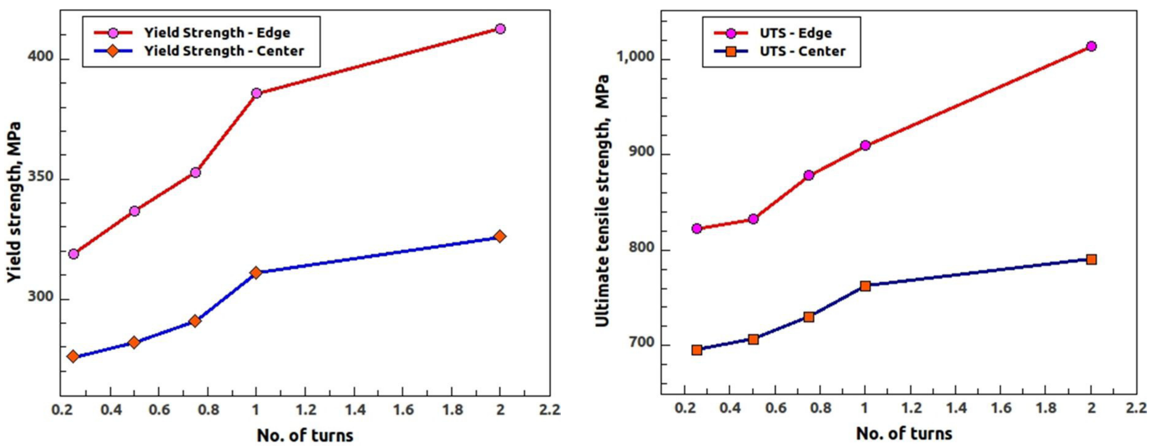
(a) (b)

Fig. 2. Variation of (a) strain hardening coefficient and (b) strength coefficient for Al-2014 alloy, processed by various numbers of HPT turns

processed number of HPT turns. Also, the rate of increment in the ultimate tensile strength is higher than the rate of increment in yield strength both at the center and circumference of disks.

The ultimate tensile strength at the circumference of disk, after 2 turns of HPT enhanced 4 times higher compared to the unprocessed sample. Similarly the yield strength at the circumference of disk, after 2 turns of HPT enhanced 2½ times higher compared to the unprocessed sample. It can be seen from Equation (2), that the basic nature of HPT process is to impose higher equivalent strain on the disk with increasing radius r. Hence, the equivalent strain is higher at the circumference of disks whereas it is lower near the center, which is very much evident from the yield strength and ultimate tensile strength values shown in Table 1.

From Fig. 2 it can be seen that the strength coefficient also increases with increasing number of HPT turns, and it is continuously higher at the circumference of the disks compared to center, for all disks processed by various number of HPT turns. However, with the increasing number of HPT turns there is decrement in the values of strain hardening coefficient, which indicates that the formability of Al-2014 alloy decreases with increasing number of HPT turns. The values of Brinell hardness number from Table. 1 also show a similar variation like the yield strength and ultimate tensile strength, which is higher at the circumference of disks and lower near the center. The general trend of microhardness variation in HPT disks is symmetrical about the disk center which being maximum at the edges decreases continuously towards the center and then again increases from the disk centers towards the other edge [Kawasaki, 2014].



(a) (b)

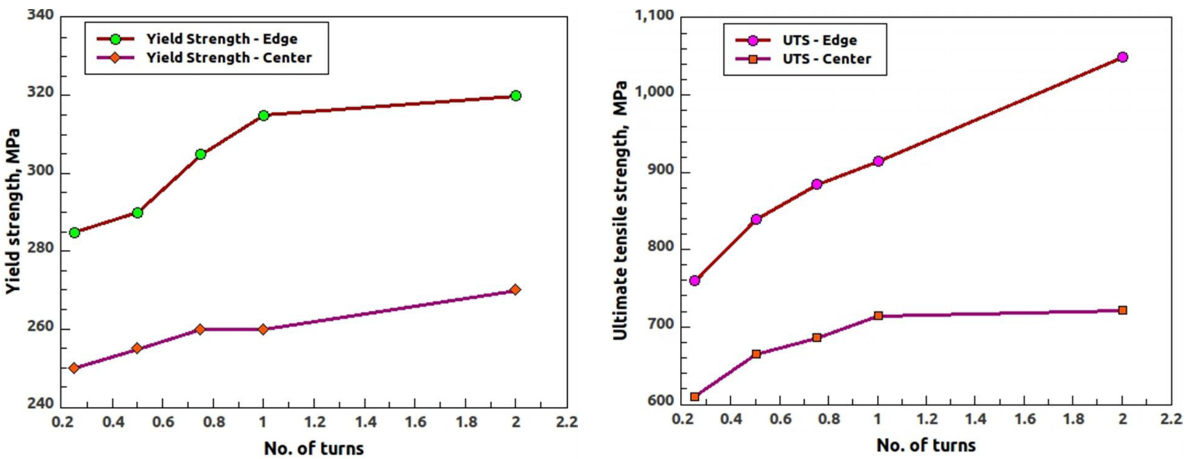
Fig. 3. Variation of (a) yield strength and (b) ultimate tensile strength for Al-2014 alloy, processed by various numbers of HPT turns

4.2. ABI technique results for HPT processed Al-7475 alloy

Table 2 ABI technique results of HPT processed Al-7475 alloy

No. of Turns	Position	K (MPa)	n	YS (MPa)	UTS (MPa)	Hardness (BHN)
1/4 turn	Center	860	0.107	250	610	144
	Edge	970	0.122	285	760	169
1/2 turn	Center	900	0.109	255	665	146
	Edge	1270	0.141	290	840	178
3/4 turn	Center	950	0.109	260	686	151
	Edge	1300	0.151	305	885	184
1 turn	Center	970	0.109	260	715	160
	Edge	1330	0.171	315	915	193
2 turns	Center	1020	0.114	270	720	162
	Edge	1760	0.196	320	1050	198

Mechanical properties of Al-7475 alloy obtained from ABI technique, for disks processed by various number of HPT turns are summarized in Table 2, both at the center and edge of disks. The yield strength and ultimate tensile strength of Al-7475 alloy show the similar trend as Al-2014 alloy which increase continuously as the number of HPT turns are increased. The ultimate tensile strength at the edge of disk after 2 turns of HPT for Al-7475 alloy attained a value of 1050 MPa where as it was 1014 MPa for Al- 2014 alloy. Fig. 4 shows the improvement of yield strength and ultimate tensile strength for Al-7475 alloy, processed by various number of HPT turns both at the center and edge of disks.



(a) (b)

Fig. 4. Variation of (a) yield strength and (b) ultimate tensile strength for Al-7475 alloy, processed by various numbers of HPT turns

5. Summary and conclusions

- Circular disks of commercial Al-2014 alloy and Al-7475 alloy were successfully processed up to a maximum of 2 turns of high pressure torsion (HPT), under an applied pressure of 6 GPa and at atmospheric temperature.
- Processing by HPT is now well-established as a procedure for the enhancement of fundamental mechanical properties including the yield strength and the ultimate tensile strength, by grain refinement to the submicrometer level or even to nanometer level. Extensive experiments on Al-2014 alloy and Al-7475 alloy have provided detailed information on the enhanced mechanical properties of these alloys.
- The yield strength and ultimate tensile strength (UTS) of the both Al-2014 alloy and Al-7475 alloy, showed a progressive improvement, with increasing number of HPT turns. The UTS at the edge of disk, after 2 turns of HPT enhanced to ~1014 MPa for Al-2014 alloy and to ~1050 MPa for Al-7475 alloy.
- Because of the smaller dimensions of HPT processed samples, it was essential to use a non-destructive testing (NDT) technique, for evaluating their mechanical properties. The automated ball-indentation (ABI) technique, requiring a very small sample area, proved to be an ideal procedure for evaluating the mechanical properties of HPT processed disks.

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