Cumhuriyet Sci. J., 43(3) (2022) 443-448
DOI: https://doi.org/10.17776/csj.952416

Cumhuriyet Science Journal

| csj.cumhuriyet.edu.tr | Founded: 2002 ISSN: 2587-2680 e-ISSN: 2587-246X Publisher: Sivas Cumhuriyet University

Comparison of Oliver-Pharr and Work of Indentation Approach to Determine the Mechanical Properties of Melt-Spun Al-12%Wt.Si-0.5%Sb Alloy

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Research Article

History
Received: 14/06/2021
Accepted: 06/09/2022

ABSTRACT

In this research, the hardness and reduced modulus of Al-%wt.12-%wt.0.5Sb melt-spun alloy were evaluated by using depth sensing indentation and atomic force microscopy techniques. We considered two approaches, Oliver-Pharr and Work of Indentation, to analyse the load-displacement curves. The ratio of final depth to maximum depth was found to be higher than the reported critical value of 0.70, which mean that pile-up was dominant in the melt-spun. A pile-up around the deformed surface was observed from atomic force microscope, which is consistent with the aferomentioned result. The hardness calculated by Oliver-Pharr method was higher than that calculated by Work of Indentation Approach. According to the results, Work of Indentation Approach was more reliable than the Oliver-Pharr approach because of reducing pile-up affect.

Keywords: Melt-spinning Al-Si-Sb alloy, Depth-sensingindentation, Atomicforcemicroscope, Oliver Pharrmethod, Work-of indentationapproach.

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Introduction

Today, Al-Si alloys are widely replacing iron based alloys in some areas such as automotive, aerospace and military industries due to its low weight, high corrosion resistance, high stiffness and moderate strength [1, 2]. Mechanical properties of Al-Si alloys can be further improved by using different methods such as rapid solidification techniques and/or addition of modifying agents. Uzun et al. have reported that hardness of eutectic Al-Si-Sb alloy produced by melt-spinning was about two times higher than those produced by induction melting and arc re-melting techniques [3]. On the other hand, the fact that one dimension of the alloy produced by melt-spinning is in the range of several microns makes it difficult to determine the mechanical properties by conventional hardness apparatus [4].

Resent years, determination of the mechanical properties of small volumes has become important issue since the mechanical properties at micro/nano-scale may differ from the bulk properties due to the size effects [5]. Indentation hardness testing is a commonly used non-destructive testing for evaluating the mechanical properties of materials. In a conventional hardness test, a fixed load is applied to a material with a diamond indenter and the dimensions of the residual indent is measured with the help of a microscope [6]. However, in many cases the size of the residual indents can be tiny to measure accurately with optical microscopy techniques.

Depth-sensing indentation (DSI) equipment developed during past two decades allows the measurement of mechanical properties of materials without the need of observation of residual indent. Beside hardness (H) and reduced modulus (Er), this technique is also capable of measurement of viscoelastic parameters, yield stress, fracture parameters, strain hardening exponent and so on [5, 7, 8]. In this technique, the displacement of indenter (h) is recorded simultaneously as applied force (P) is loaded and unloaded in a specimen. Once a load-displacement (P-h) curve is obtained, H and Er of a specimen can be calculated using different empirical models proposed in literature. The two most well-known models, Oliver-Pharr and Work of indentation, will be described in detail in Section 2.

During the indentation of a material, deformations may occur at the edges of residual indent. This kind of deformations are agglomeration to outward and collapsing to inward of the indent edges, which are called pile-up and sink-in, respectively. Pile-up and/or sink-in can seriously affect the true hardness of material if they are not taken into account in calculations [9]. This kind of deformations can be directly observed by atomic force microscopy or indirectly determined by empirical values obtained from P-h curve.

It is very important to determine the mechanical properties of melt-spun alloys correctly from P-h curves. Therefore, in this research, we studied how the true hardness and reduced modulus can be calculated from P-h curves using two models and compared them in terms of pile-up effect.

Theoretical Background

Figure 1 shows a schematic load-displacement (P-h) curve and resultant indent profile of an ideal material response. Two well-known mechanical properties, namely hardness (H) and reduced modulus (Er), can be calculated by analyzing P-h curve.

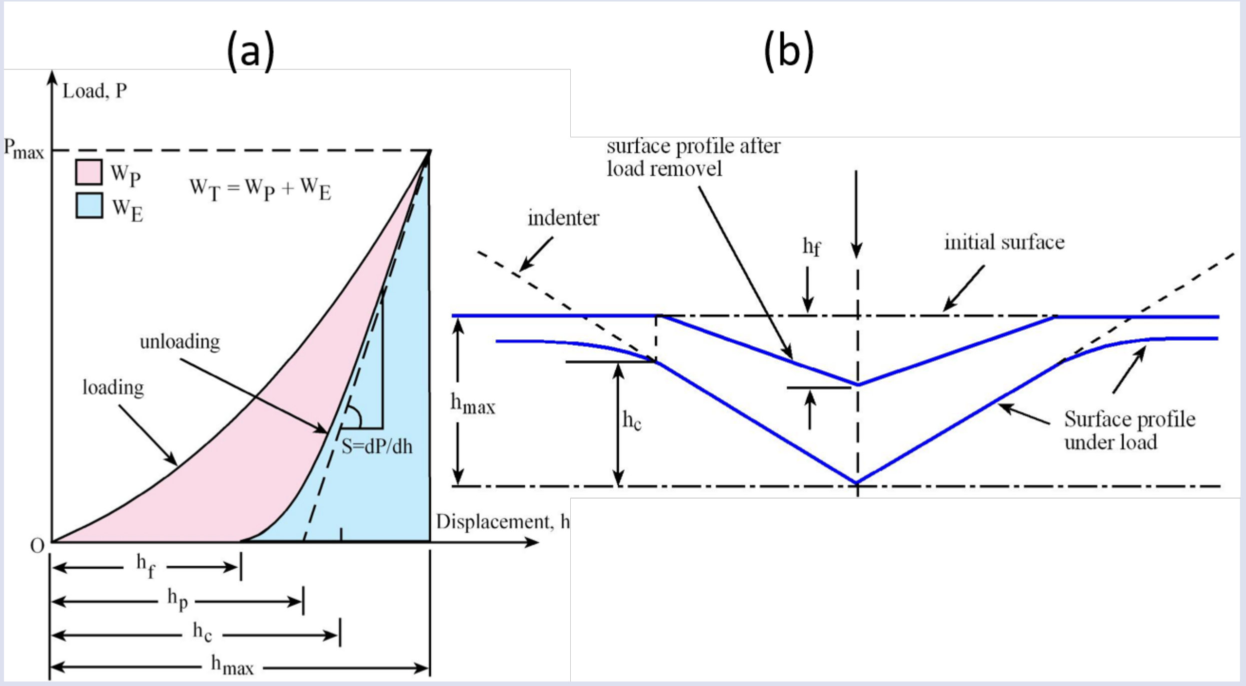

Figure 1. a) A schematic load-displacement curve, b) A schematic illustration of an ideal material response to indentation loading/unloading and resultant profiles. The abbreviations, hmax, hf, hp and hc are total penetration depth, final residual depth, plastic depth, and contact depth, respectively.

Oliver-Pharr (OP) Method

Oliver and Pharr (OP) method is the most adopted method in literature [10]. In this method, firstly unloading curve fits to the power-law relation:

$$P = K\left(h_{\max} - h_f\right)^t \qquad (1)$$

where *P* is the indentation load, *K* and *t* are the fitting parameters, h_f is the final depth. The stiffness *S* can be obtained by

$$S = tK\left(h - h_f\right)^{t-1} \qquad (2)$$

The contact depth h_c can be estimated using:

$$h_c = h_{\max} - \varepsilon \frac{P_{\max}}{S} \qquad (3)$$

where *ε* is a constant related to an indenter geometry and 0.72 for conical indenters. Finally, the indentation hardness H_{OP} and reduced modulus *Er* can be determined by

$$H_{OP} = \frac{P_{\max}}{A_c} \qquad (4)$$

$$Er_{OP} = \frac{S}{2}\sqrt{\frac{\pi}{A_c}} \qquad (5)$$

where A_c is the contact area equal to $24.5h_c^2$ for Vickers indenter.

Work of Indentation Approach (WIA)

Work of indentation approach is another analysis method which has been extensively studied since it was first proposed by Stilwell and Tabor [11]. The method has been improved by Sakai [12] who put forward a relationship between the energy of the hysteresis indentation loop and the hardness. Attaf [13] used the work of indentation approach to suggest some energy terms and Tuck [14] developed a simple formula by using the relation between work of indentation and Vickers hardness number. This method describes indentation experiment process as the use of the energy dissipated or work done during the indentation. The energies can be easily calculated thorough integrals. For instance, total work (W_T) and elastic work (W_E) are the under the areas of loading curve and unloading curve, respectively. In this case, plastic energy is equal to the difference between other energies (Figure 1a) [14, 15].

$$W_P = W_T - W_E \qquad (7)$$

Tuck et al. [14] proposed a fallowing hardness formula based on plastic work of indentation:

$$H_{WIA} = \frac{\kappa P_m^{\,3}}{9 W_P^{\,2}} \qquad (8)$$

where χ is a constant equal to 0.0378 for Vickers indenter. Reduced modulus can be determined using elastic and total work of indentation as follow equation [16];

$$\frac{W_E}{W_T} = 5\left(\frac{H_{WIA}}{Er_{WIA}}\right) \tag{9}$$

Materials and Methods

Al-wt.%12Si-wt.%0.5Sb alloy from high purity of elements (Al: %99.9, Si: %99.999, Sb: 99.999 wt.%) was first melted in a graphite crucible using induction furnace and then re-melted five times to ensure homogeneity. The melt-spun alloy was produced using a melt-spinner apparatus (Edmund Bühler) with a rotating speed of 40 m/s. As-received melt-spun was 1 cm in wideness and 25 µm in thickness. Before indentation test, the surface of melt-spun was polished to 0.25 µm using diamond lap wheels. Load-displacement curves were obtained from depth sensing indentation instrument (Shimadzu, DUH-W201S) with the load and displacement resolutions of ±19.6 µN and ± 1 nm, respectively. Different loads ranging from 200 to 1200 mN were applied with a loading rate was 23,4 mN/s. AFM analysis were performed at non-contact mode for a scanning area of 80x80 µm^2.

Results and Discussion

Figure 2 shows microstructure of melt-spun alloy. The microstructure consists of fully equiaxed α-Al grains and homogeneously distributed fine fibrous Si eutectics (Figure 2a). Moreover, Si spheres in nano-scale size are dispersed in α-Al (Fig 2b). The fine and homogenous microstructure of Al-Si alloys is attributed to fast cooling rate, which is typical for melt-spun alloys. Similar results have been reported in literature [3, 4, 17, 18].

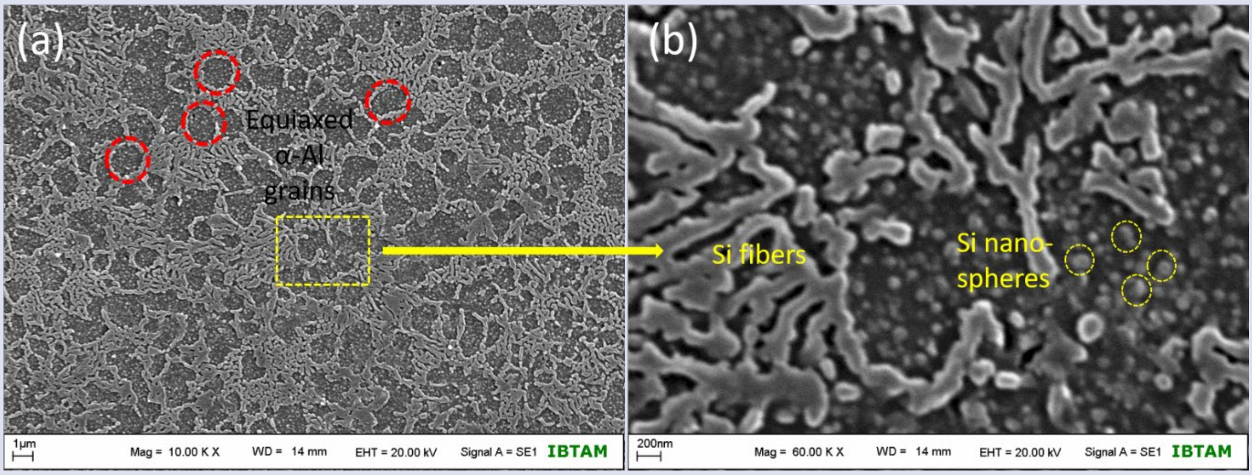

Figure 2. SEM image shows the microstructure of melt-spun ribbon. (a) homogenously distributed equiaxed cellular α-Al grains, (b) fine silicon fibers and nano-scale silicon spheres in α-Al.

Figure 2 SEM image shows the microstructure of melt-spun ribbon. (a) homogenously distributed equiaxed cellular α-Al grains, (b) fine silicon fibers and nano-scale silicon spheres in α-Al.

3D profile and corresponding profile section of indented area in Figure 3a clearly shows a pile-up along the four edges of the indentation imprint. The schematic illustration of pile-up phenomena is depicted in Figure 3b, which shows a difference between apparent and true contact diameter. When a pile-up is dominant at the around of deformed surface, an accuracy of hardness and reduced modulus are effected since the apparent contact depth will be smaller than true contact depth as depicted in Figure 3b. Contact depth after force removal can easily be scaned by AFM. The apparent and true contact depths are measured as 2.23 and 1.89 µm from Figure 3a. The hardness calculated using Equation 4 is approximately 2120 and 1530 MPa for apparent and true contact depths, respectively. These results imply that a pile-up seriously affects the true hardness. Hardness can precisely determine by AFM technique, but this technique usually takes a lot of time.

Apart from AFM technique, the ratio of final depth to maximum depth (h_f/h_{max}) is an easy measurable parameter from depth sensing indentation data that can be used to identify pile-up or sink-in behavior of a tested material. h_f/h_{max} values varying from 0.71 to 0.80 for different loads are listed in Table 1. According to Bolshakov and Pharr [19], 0.70 for h_f/h_{max} is a critical value and above this value, pile-up becomes significant for tested materials. As can be seen from Table 1, the values of h_f/h_{max} are higher than the reported critical value for each load. This result indicates the existence of pile-up around the indents, which is in agreement with AFM observations.

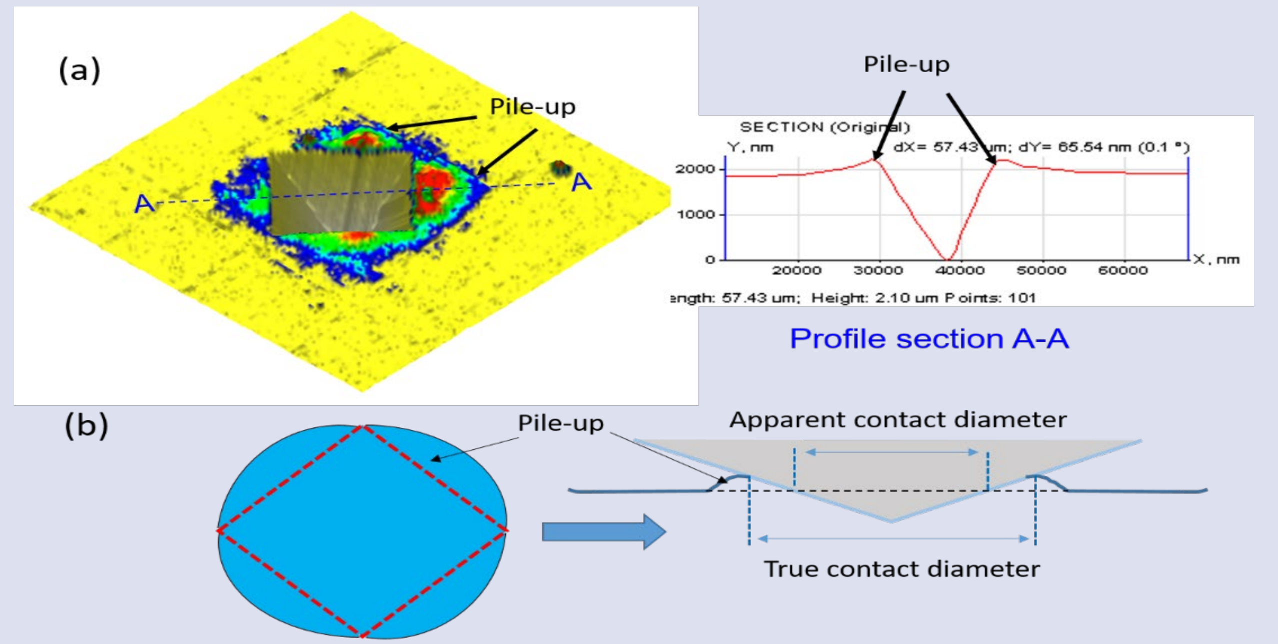

Figure 3 (a) AFM 3D image of indented surface shows significant pile-up deformation near the indented surface and cross-sectional profiles of the indent with applied load of 200 mN, (b) Schematic illustration of pile-up behavior.

In order to determine the H and Er of the melt-spun alloy, a series of load-displacement (P-h) curves were obtained from DSI test. An overlapping of loading curves means that the material is homogenous microstructure, which is compatible with SEM image (Figure 2). To obtain H and Er, P-h curves were analyzed two well adopted approaches, Oliver-Pharr (OP) and work of indentation (WIA), which are described above in detail. Figure 5 shows the *H* and *Er* values as a function of peak loads. From the figure, one can see that H and Er values exhibit load dependent behavior, namely they decrease with increasing applied load. This phenomenon is called indentation size effect and observed different kinds of materials, such as metallic alloys, single crystals, ceramics, superconductors, polymers, etc. [20-22]. ISE can exist in some situations, such as work hardened surface, surface oxides, tip bluntness or poor tip-shape calibrations. Many models have been proposed in the literature to calculate load independent hardness. Perhaps the most reasonable model explaining the physical reasons behind this phenomenon was proposed by Nix and Gao [23]. This approach is based on geometrically necessary dislocations and successful to explain ISE in metallic alloys. Analysis of ISE by different models is not the object of this study, so one can found more detailed information in elsewhere [21].

From Figure 5, it is clearly seen that the hardness values calculated from OP (H_{OP}) higher than that of WIA (H_{WIA}). It is well known that the OP method does not take into account the pile-up effect observed in materials [24]. The contact depth, hc, is therefore greatly underestimated in the OP method, so overestimated hardness values are obtained [14]. We therefore suggest that the H_{WIA} values are more convenient than the H_{OP} values for our sample. Moreover, the hardness calculated from AFM (1530 MPa) is close to H_{WIA} (1650 MPa) than H_{OP} (1850 MPa) for applied load of 200 mN. AFM results further confirm the reliability of WIA. Reduced modulus, Er, of the sample is given in Figure 5 b. Unlike the H values, Er values calculated with two methods are found to be very similar to each other. This may be explained by the fact that the pile-up behavior is related to plastic deformation rather than elastic deformation. Since hardness is defined as resistance to plastic deformation, it is more likely to be affected by pile-up.

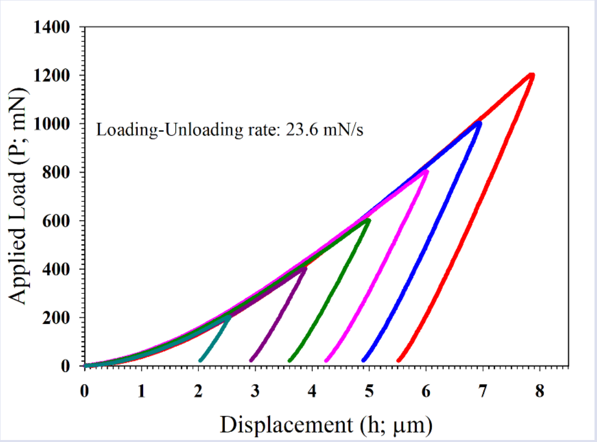

Figure 4. A series of load-displacement plots at different peak loads, ranging from 200 mN to 1200 mN.

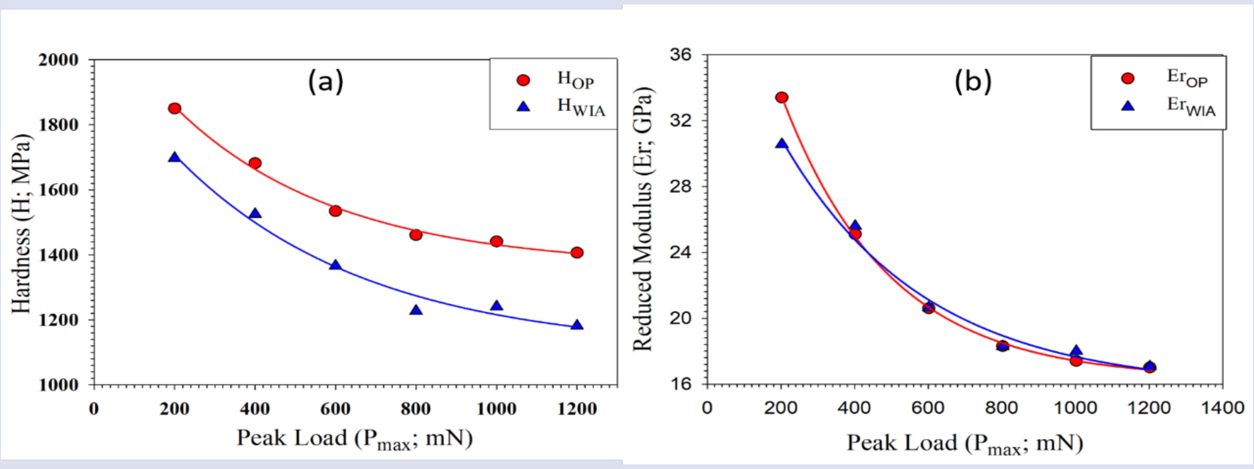

Figure 5 a) Hardness and b) Reduced modulus variation as a function of the peak load.

Conclusion

In this study, we aimed to investigate the mechanical properties of melt-spun Al - wt.%12Si – wt.%0.5Sb alloy by using depth-sensing indentation technique and compared two well-known approaches, Oliver-Pharr and work of indentation, in order to find the most suitable one. The ratios of h_f/h_{max} was found to be higher than critical value of 0.70 at various peak loads, which mean that a pile-up affect was dominant in our sample. The pile-up was further confirmed by AFM observation. Hardness and reduced modulus of the sample were calculated using Oliver-Pharr and work of indentation approaches. It was found that the values obtained by work of indentation approach were more reliable than the Oliver-Pharr approach because of reducing pile-up effect.

Conflicts of Interest

No conflict of interest was declared by the authors.

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