Fabrication and Characterization of Bulk Nanocrystalline Layer on the Aluminum 6061 Surface by Shot Peening (SP)

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Abstract

The fabrication of bulk nanocrystalline (NC) layers on aluminum alloy Al 6061-T6 using shot peening was investigated in this paper. The cross-sectional microstructure of the samples showed a sharp reduction of grain size to nano scale in the near surface. The NC layers were characterized using optical, scanning electron microscopy (SEM), transmission electron microscopy (TEM), microhardness and nanohardness measurements. 2-dimensional (2D) surface topography of the peened surface was also carried out to measure surface roughness. Roughness of the peened surfaces was dependent on the hardness of sample materials. NC layers of 20 μ m to 50 μ m thickness were measured using optical and SEM images for the two types of aluminum alloys. The SEM images clearly showed the demarcated NC layers. The nanohardness measurements of the aluminum alloys showed that the NC layers had much higher hardness than the bulk material. The microhardness measurements also confirmed the mentioned phenomena. A TEM study was carried out on the NC samples and the grain size was about 30 to 40 nm.

Keywords: Bulk nanostructured material, nanomanufacturing, surface integrity, shot peening, aluminum alloy.

Introduction

It is a well known fact that a smaller grain size in metals enhances its' mechanical properties like strength, toughness, hardness and wear resistance. Consequently great efforts have been made in industry to refine grains of materials. Some of the methods used for grain refinement include equal channel angular pressing (ECAP) [1-3], accumulative roll bonding (ARB) [4-5]. By these processes grain refinement to sub micron level is possible. However, to obtain a grain size smaller than 100 nm, methods like consolidation of ultrafine powders [6], electrodeposition [7], and severe plastic deformation [8] have been used. Out of these, severe plastic deformation processes have the advantage of producing mass scale nanostructures in an economically viable way as it uses the "top down" approach rather than the traditional "bottom up" approach. Severe plastic deformation processes include ball milling [9-11], high pressure torsion [12], ball drop test [8], ultrasonic shot peening [13-14], and shot peening (SP) [15-17].

Surface nanocrystallization of aluminum alloys has been reported by several studies such as grain size of 20 to 100 nm on 1420 aluminum alloy by high energy shot peening [18], about 50 nm in the surface layer of 7A04 aluminum alloy by circulation rolling plastic deformation (CRPD) [19], and about 30–40 nm on 7050 aluminum alloy by High Pressure Shot Peening (HPSP) [20]. The objectives of this research are several fold: (i) to study the feasibility of fabrication of bulk nanostructured structures on AL 6061-T6 alloy using shot peening (SP); (ii) to

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investigate mechanical properties of the nanostructured layer; and (iii) to study the effects of peening parameters on the nanostructured layer.

Experimental Setup

SP testing conditions are in Table 1. The first type shot SBM300 is cast iron shot with chemical composition C 1%, Mn<0.35~1.0%, Si>0.40% and size of 50-125 μ m. The second type of shot is a stainless aluminum alloy shot, JIS SUS304, with chemical composition C < 0.08%, Mn<2.00%, Si<1.00% Ni 8.5-10.5%, Cr 18-20% and size of 300 μ m. Peening was done on the central part the samples till required coverage was achieved.

Material	Coverage (%)	Shot	Pressure
AI 6061	1000		
	2000	SBM300	0.2 MPa
	3000	(50µm)	0.2 IVIFa
	4000		
	1000		
	2000	SUS304	0.1 MDo
	3000	(300µm)	0.1 WFa
	4000		

Table 1Peening parameters for samples

After the peening, the surface topography was characterized using a profilometer. The samples were then cut in the middle of the peened zone to characterize the cross section. The microstructure of the cross section was obtained by optical and scanning electron microscope. The nano hardness of the NC layers and the bulk of the material were measured by a Hysitron nanoindenter using suitable loads. In addition, the microhardness was measured using a Vickers indenter at a load of 10 g. Finally electron transparent TEM foils were made using standard procedure.

Results and Discussions *Effect of coverage*

To study the effect of coverage, AI 6061 samples were peened using two kinds of shots at four coverages ranging from 1000% to 4000% as seen in Table 1. The other parameters i.e. the shot size, the peening pressure, and the peened material remain unchanged for a given test. After experiments. the samples have the been characterized study surface to topography, microstructure, hardness and nanohardness and the results are summarized in the following paragraphs.

<u>Surface roughness</u>: Figure 1 shows effect of coverage on the surface roughness. As the coverage is increased from 1000 % to 4000 % for





the 50 μ m shot, the roughness value steadily increases from a value of 3.99 μ m to 6.71 μ m. This trend suggests that the roughness value bears almost a linear relation with the peening

coverage. From a physical point of view, it may be stated that prolonged and repeated impacts seem to drive the surface roughness higher and higher. Another observation regarding the profile is that the numbers of peaks increase as the coverage increases i.e. the surface becomes more peaked with increase in coverage. Also the peak to valley distance reduces as the coverage is increased. This phenomenon occurs because of the repeated impact on the surface on account of higher coverage. The repeated impact make the surface more peaked. For the samples peened with 300 μ m shot, the surface roughness remained around 4.8 μ m for the coverage values up to 3000%. However, at 4000 %, the measured value reached about 6.6 μ m. The larger diameter of the shots may responsible for such behavior.

<u>Microstructure:</u> Figure 2 shows the effect of coverage on evolution of the NC layers formed in Al 6061 peened with 50 μ m shots at a pressure of 0.2 MPa. The nanocrystalline layers are very uniform and thick regardless of the peening coverage. We also observe that as the coverage is increased from 1000 % to 4000 %, the thickness of NC layer increases slightly. At coverage of 1000% the thickness of the NC is about 20 μ m whereas at a coverage of 4000 it only increased to a value of about 30 μ m. The morphology of NC layer remains unchanged with increased coverage. Even at 4000% coverage, the NC layer was continuous and devoid of cracks. This may be attributed to the high ductility and malleability of aluminum.

Figure 3 shows the effect of coverage on the evolution of the NC layers formed in Al 6061 peened with 300 μ m shots at a pressure of 0.1 MPa. In contrast with the nanocrystalline layers formed by the 50 μ m shots, the NC layers formed by the 300 μ m shots appear to have a thickness variation. At the same time, their average thickness - about 45 μ m - is much greater than those formed by the 50 μ m shot. We also observe that as the coverage is increased from 1000 % to 4000 %, the thickness of NC layer increases significantly. At coverage of 1000% the thickness of the NC is about 25 μ m whereas at a coverage of 4000 it doubled to a value of about 50 μ m. The morphology of NC layer shows severe plastic deformation of the metal and remains unchanged with increased coverage. As compared to the NC layer obtained by the 50 μ m shot, the NC layer fabricated by the 300 μ m shot shows distinct circular patterns. This may be attributed to the larger size of the shot and higher energy of impact.





1000 %

Figure 2. Effect of coverage on microstructure – Al 6061 50µm shot

Figure 3. Effect of coverage on microstructure – Al 6061 300µm shot

2000%

50 µm

50 um

50 µm

<u>Microhardness</u>: Table 2 shows the effect of coverage on the microhardness of Al 6061 peened with 50 μ m and 300 μ m shots at a pressure of 0.2 MPa and 0.1 MPa respectively. The representative corresponding SEM images of the dents are shown in Figure 4. We find that for all coverage values the hardness measured in the NC layer was higher than in the bulk material. The hardness values have been measured in the NC layer, just below the NC layer and in the

deep subsurface (bulk material). The hardness in the NC layer is the highest, and the hardness just below the NC layer and in the deep subsurface are about the same. This trend is observed for all the peened samples.

	Microhardness HV (10 g mass)		
Shot	size 50 µm	Shot s	size 300 µm
Coverage	Microhardness	Coverage	Microhardness
	210 (NC)		159(NC)
1000	110 (Below)	1000	119(Below)
	127 (Bulk)		110(Bulk)
	193(NC)		129(NC)
2000	102(Below)	2000	119(Below)
	100(Bulk)		102(Bulk)
	234(NC)		185(NC)
3000	119(Below)	3000	135(Below)
	117(Bulk)		110(Bulk)
	219(NC)		182(NC)
4000	110(Below)	4000	111(Below)
	115(Bulk)		110(Bulk)

Table 2. Microhardness



Figure 4. SEM of the microhardness dents

The small grain size and the severe plastic deformation contribute to the highest hardness of the NC layer whereas the large grain size causes the hardness in the subsurface to be significantly lower. It is observed that as the coverage is increased from 1000 % to 4000 % the hardness remains in the same range considering statistical variation.

For the samples peened with the 300 µm shot, the hardness shows an increasing trend in general. For the coverage value of up to 2000 % the average hardness is 140 where as for coverage from 3000 to 4000 have an average hardness of 180. The hardness values measured in the NC layers are higher than the bulk.

Nanohardness: The effect off coverage on the nanohardness shown in Table 3 shows that the NC layers have a higher hardness than the bulk material in general. The hardness value decreases as the depth below the surface is increased. Since the scale of measurement is very small, the difference in the hardness between NC layer and the bulk material is not as high as the microhardness. However the overall trend is similar to the microhardness measurement.

Effect of energy of impact

The energy of impact plays a crucial role in formation of NC layers. To study

	Table	3.	Nanohardness
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Nanohardness (GPa)			
Shot s	size 50 µm	Shot s	ize 300 µm
Coverage	Nanohardness (GPa)	Coverage	Nanohardness (GPa)
	1.99(NC)		2.35(NC)
1000	1.80(Below)	1000	2.21(Below)
	2.00(Bulk)		2.10(Bulk)
	2.63(NC)		1.85(NC)
2000	1.80(Below)	2.00(Bulk) 2.63(NC) 1.80(Below) 2.05(Bulk) 2.42(NC) 2.15(Below) 3000	1.49(Below)
	2.05(Bulk)		1.83(Bulk)
	2.42(NC)		2.80(NC)
3000	2.15(Below)	3000	1.85(Below)
	2.30(Bulk)		2.20(Bulk)
4000	2.53(NC)	4000	4.05(NC)
	2.03(Below)		2.10(Below)
	2.43(Bulk)		2.12(Bulk)

this effect, two shot sizes have been peened at different pressures. As per Table 1 the shot sizes are 50 μ m and 300 μ m, respectively, and the corresponding pressures are 0.2 MPa and 0.1 MPa, respectively.

<u>Microstructure</u>: Figure 5 shows the effect of impact energy on the microstructure of the peened 6061 samples. In the 6061 sample the NC layer corresponding to 50 μ m shot is about 25 μ m thick where as for the 300 μ m shot it is of about double thickness. This higher thickness may be produced due to the higher impact energy of the 300 μ m shot. This higher impact energy causes the thickness of the NC



Figure 5. Effect of impact energy on microstructure

layer to be much higher than produced by the 50 μm shot.

The difference between the two peened surface layers is that the 50 μ m shot produces a continuous layer whereas the 300 μ m shot produces an NC layer which is interrupted in nature.

<u>Microhardness</u>: Table 4 shows the hardness values corresponding to the impact energy effect. The smaller shot size of 50 μ m yield a higher hardness as compared to 300 μ m shot size. This is because of the higher impact velocity.

<u>Nanohardness</u>: Table 4 also shows the nanohardness values corresponding to the impact energy effect. The smaller shot size of 50 μ m yield a lower hardness of 2.53 GPa as compared to the 300 μ m shot size which gives a hardness of 4.05. Since nanohardness is a very localized phenomenon there seems to be an anomaly when compared with the microhadness readings.

Impact energy Effect (AI 6061)			
Pressure	Microhardness (10 g)	Pressure	Nanohardness
	219(NC)		2.53(NC)
0.2 MPa(50µm shot)	110(Below)	0.2 MPa (50µmshot)	2.03(Below)
	115(Bulk)		2.43(Bulk)
	182(NC)		4.05(NC)
0.1 MPa (300µm shot)	111(Below)	0.1 MPa(300µm shot)	2.10(Below)
	110(Bulk)		2.12(Bulk)

Table 4. Impact energy effect on microhardness: 50 μ m vs. 300 μ m

Grain Size

A TEM microscopy study has been conducted in order to confirm the average grain size of the material. Figure 6 shows the comparison of the grain size of the bulk vs. the nanocrystalline layer. From the grain size of the bulk material it is clearly seen that the grain size is in the order of about 3 μ m. However after shot peening, the grain size is about 30 to 40 nm as seen in the Figure. The dark field image on the right show equiaxed grains of the order of



Figure 6. TEM micrographs of unpeened vs. peened

30nm. This demonstrates that the layer at the top surface is comprised of nanocrystalline grains. This is why the hardness of the zone is greater than the bulk material as has been described above.

Conclusions

The key findings of the research are summarized as following

• The SP process is an effective way to produce bulk nanocrystalline layers of Al 60661-T6 alloy with the average grain size of 30 - 40 nm.

• The 50 μ m shots tend to produce thick and continuous nanostructures where as the 300 μ m samples for thick but intermittent NC layers.

• A smaller shot peened at a higher pressure produces a distinct NC layer whereas the larger shots produce a thicker distinct NC layer which is separated.

• All the NC layers have a higher hardness than the bulk material as measured by microhardness and nanohardness.

References

[1] R.Z. Valiev, N.A. Krasilnikov, N.K. Tsenev, Mater. Sci. & Eng., A137 (1991) 35-40.

[2] R.Z. Valiev, I.V. Alexandrov, R.K. Islamgaliev, Nanocrystalline materials: science and technology, In: Chow GM, Noskova NI, editors., Nato ASI. Dordrecht: Kluwer Academic Publishers, 1998 pp.121.

[3] R.Z. Valiev, O.A. Kaibyshev, R.I. Kuznetsov, R.Sh. Musalimov, N.K. Tsenev, DAN SSSR; 301(4) (1988) 864.

[4] Y. Saito, N. Tsuji, H. Utsunomiya, T. Sakai, R.G. Hong, Scripta Materialia 39/9 (1998) 1221-1227.

[5] Y. Saito, H. Utsunomiya, N. Tsuji, T. Sakai, Acta Materialia 47/2 (1998) 579-583.

[6] H. Gleiter, 2nd Riso Int. Sym. Metall. Matl Sci. eds, N Hansen, A Horsewell, and H Lilholt, (1981) pp.15-21.

[7] G. Palumbo, S.J. Thorpe, K.T. Aust, Scripta Metallurgica et Materialia 24(7): (1990)1347-1350.

[8] M. Umemoto, K. Todaka, K. Tsuchiya, Materials Science and Engineering A 375–377 (2004) 899–904.

[9] J.S.C. Jang, C.C. Koch, Scripta metallurgica et materialia, 24(8) (1990)1599-1604.

- [10] H.J. Fecht, E. Hellstern, Z. Fu, W.L. Johnson, Mater. Sci. & Eng., 21A(9) (1990) 2333-2337.
- [11] Y. Todaka, M. Nakamura, S. Hattori, K. Tsuchiya, M. Umemoto, Mater. Trans., 44 (2003) 277- 284.
- [12] R.Z. Valiev, Yu.V. Ivanisenko, E.F. Rauch, B. Baudelet, Acta Materialia, 44(12) (1996) 4705-4712.
- [13] N.R. Tao, M.L. Sui, J. Lu, K. Lu, Nanostructured Mater., 11 (1999) 433- 440.
- [14] F.A. Guo, N. Trannoy, J. Lu, Superlattices and Microstructures 35 (2004) 445-453.
- [15] I. Altenberger, B. Scholtes, U. Martin, H. Oettel, Mater. Sci. & Eng., 264 (1999) 1-16.
- [16] X.Y. Wang, D.Y. Li, Wear, 255 (2003) 836-45.
- [17] Y. Todaka, M. Umemoto, Y. Watanabe, K. Tsuchiya, Mater. Sci. Forums, 503-504 (2006) 669- 674.
- [18] L. Hu, K. Wang, G. Liu, B. Xu, Trans. Nonferrous Met. Soc., China, 15(3) (2005) 615-618.

[19] H. Ye, X. Fan, Trans. Nonferrous Met. SOC. China, 16 (2006) 656-660.

[20] Y. Mai, X. Jie, L.L. Liu, N. Yu, X.X. Zheng, App. Surface Sci., 256 (2009) 1972-1975.