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Powder Metallurgical 6061Al Nano/Micro Al_2O_3 Particulate Reinforced Composites- A Comparative Study

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ABSTRACT
In this study, mechanical alloying (MA) technique was used to disperse 10 wt% of Al_2O_3 nano/micro particles in 6061 Al powders using the high energy mill. MA was carried out for the period up to 6 h in nitrogen atmosphere. XRD of mechanically processed powder was carried out to assess grain size in powder. The processed powder was then degassed and consolidated by hot pressing. The composites thus prepared were characterized for their microstructure using image analyzer and electron microscopy. The nanocomposites have substantially higher hardness than the micro composite. The tensile strength of the nanocomposite exhibited inverse Hall-Petch relationship for the cases of mechanical alloying period exceeding 2h. However, the bimodal composite was found to have a moderate combination of the tensile strength, ductility and hardness.

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Introduction

Composite materials that traditionally incorporate micron scale reinforcements in a bulk matrix offer opportunities to tailor material properties. With the advent of nanomaterials, nanocomposites are envisioned, and are being developed, with properties such as hardness, tensile strength, ductility, density, thermal and electrical conductivity, and wear resistance that overcomes the limitations for metals or composites that contain micron scale reinforcements [1]. In addition, exceptionally high thermal conductivities, possible in selected nanocomposites, will find applications in thermal management applications in computers [2]. Metal matrix nanocomposites can be designed to exhibit high thermal conductivity, low density, and matching coefficient of thermal expansion with ceramic substrates and semiconductors, making them ideal candidates for such applications.

Aluminum nanocomposites are predicted to surpass the weight reduction currently realized through the use of polymer-based nanocomposites and polymer-based fiber composites in aerospace applications primarily because these metal matrices have higher strength and stiffness. They also have much better thermal stability [3-4].

The development of metal matrix nanocomposites (MMNCs), however, is still in its infancy. As with conventional metal matrix composites with micron-scale reinforcements, mechanical properties of MMNCs are strongly dependent on the properties of reinforcements, distribution, and volume fraction of the reinforcement, as well as the interfacial strength between the reinforcement and the matrix [5]. In addition, due to their high surface area and surface dominant characteristics, these materials may also be highly reactive in metal matrices [6]. Because of these concerns, processing methods are being developed to produce MMNCs with uniform dispersion of nanomaterials and little deleterious interfacial reactions.

The methods that have been used to synthesize metal matrix nanocomposites include powder metallurgy, deformation processing, vapor phase processing, and in some cases solidification processing. Powder metallurgy involves the preparation of blends of powders of metal and reinforcements, followed by consolidation and sintering of the mixtures of powders to form the part.Density of the components can be controlled easily through powder metallurgy method when compared to other manufacturing processes [7-8]. When the reinforcement in a metal matrix is brought down from micron-scale to nano-scale, the mechanical properties are often substantially improved over what could be achieved using micron-scale reinforcements. In some cases, the nanoscale reinforcement leads to property changes in the matrix itself. For instance, nanoscale reinforcements can lead to nanosize grains in the matrix, which will increase the strength of the matrix.

Though nanocomposite materials exhibit ultra high-strength, there is often a trade-off that results in decreased ductility. In the case of nanostructured grains, the presence of hard precipitates or nanoparticles in a metal matrix may act to initiate, drag and pin dislocations, reducing dynamic recovery, and thus resulting in a high strain-hardening rate that, in turn, produces larger uniform strains and higher strengths in the MMNCs, along with higher ductility [9].

There are exciting opportunities for producing exceptionally strong, wear resistant metal matrix nanocomposites with acceptable ductility. Metal matrix nanocomposites can lead to significant savings in materials and energy and reduce pollution through the use of ultra-strong materials that exhibit low friction coefficients and greatly reduced wear rates.

Mechanical alloying (MA) is a widely accepted technique for synthesizing nanocomposites. It is one of the simple and useful techniques for attaining a homogeneous distribution of the inert fine particles within a fine grained matrix [10]. The MA process consists of repeated cold welding,

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fracturing and re-welding of powder particle mixture in a high-energy ball mill [11]. Owing to the characteristic processes of attrition ball mill which involves repeated deformation, cold welding and fragmentation, structural changes like decrease in the crystallite size and the accumulation of lattice strain occurred in severely deformed powders. The reduction in the crystallite size of powder is induced by the formation of large amount of linear defects particularly dislocations which result in the formation of high dislocation density regions in the grains, piling up of the grain boundaries or irregular clusters into the grains [12]. High energy ball milling can be used to improve reinforcement distribution throughout the matrix, because deformation, fracturing and cold welding of the powder occur, giving riseto the reinforcing particles being well embedded into every aluminum particle [13].The severe plastic deformation of powder particles can lead to grain refining, accumulation of internal stress, change of the lattice parameter and formation of cell structure [14].
For the present study, 6061Al matrix was selected to disperse Al_2O_3 nano/micro particles. For achieving a uniform distribution of the reinforcement phase in the matrix, MA technique was used and the resultant powder was consolidated by hot pressing. Aim of these experiments was to study the effects of MA duration and size of reinforcement particles on mechanical behavior of the composite.

Experimental

Details of the raw materials used in the present investigations are as given in Table 1. The morphology of as-received powders was analyzed using field emission scanning electron microscope (FESEM). 6061Al powder was spherical and rounded irregular, Al_2O_3 (10μm) powder was rounded irregular, and Al_2O_3 (50nm) powder was spherical and rounded as shown in Fig.1.

Table 1: Details of the raw materials

S.No	Material	Average Particle Size	Supplier
1	6061 Al powder (atomized)	70 μm	ECKA Granulate Velden GmbH, Germany
2	Al_2O_3 powder	10 μm	Sigma Aldrich
3	Al_2O_3 powder	50 nm	Sigma Aldrich

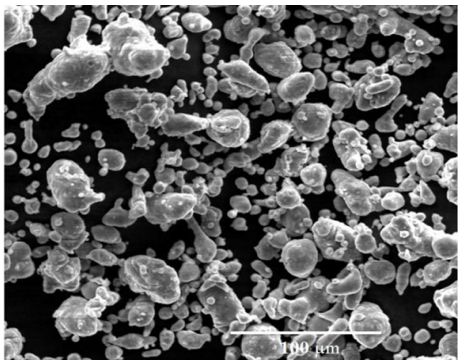
The 6061Al matrix powder, along with 10wt% of Al_2O_3 nano/micro particles, was milled in Szegvari attrition mill at a rotation speed of 350 rpm. Milling time was varied from 2-6 h. Mechanical milling beyond 6 h was found to make the resultant powder pyrophoric. The milling media used was hardened steel balls of 10 mm diameter. The weight ratio of milling media to powder was kept 10:1. Milling was carried out in purified nitrogen (99.99 % pure) atmosphere to prevent oxidation during milling. The MA powders were then degassed at 200^{0}C under a vacuum of 10-2torr. To avoid any unwanted and excessive cold welding of powder particles amongst themselves, onto the internal surfaces of the vial and to the surface of the grinding medium during milling, 1.0 wt% Acrawax C was used as process control agent (PCA).

A bimodal mixture of MA(6h) 6061Al - 10wt% Al_2O_3 (50nm) powder and Y-cone mixed 6061Al-10wt%Al_2O_3 (50nm) powder, in the ratio of 1:1, was also prepared by mixing these powders in Y-cone mixer for 2h. This mixture was prepared to develop bimodal composite having both the ductile and hardened (due to MA) matrix.

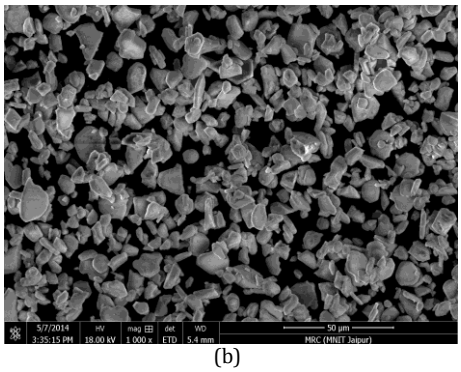
All the powder samples prepared were consolidated by hot pressing at 460^{0}C under a constant pressure of 120 MPa for 30 min. Hot-pressed specimens of 40mm x 10mm x 5mm were prepared. The details of all powder samples/ hot pressed specimens prepared are shown in Table 2.

Table 2: Details of powder samples/ hot pressed specimens

Specimen no.	Details of hot pressed specimen
1	6061 Al powder (as received)
Nanocomposite	
2	MA(2h) 6061Al-10wt% Al_2O_3 (50nm)
3	MA (4h) 6061Al-10wt% Al_2O_3 (50nm)
4	MA(6h) 6061Al-10wt% Al_2O_3 (50nm)
Microcomposite	
5	MA(6h) 6061 Al-10 wt% Al_2O_3(10 μm)
Bimodal composite	
6	MA(6h)+Y cone mixed (1:1 ratio)



(a)


(b)

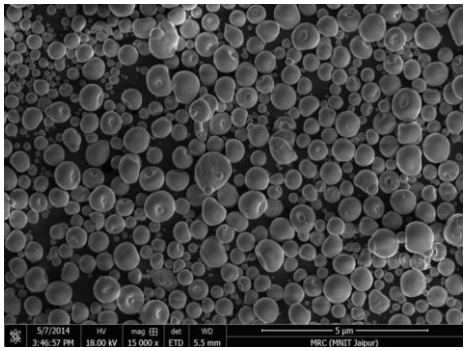

(c)

Figure 1: SEM micrographs of: (a) as received 6061Al powder; (b) Al_2O_3 powder (10 μm); (c) Al_2O_3 powder (50 nm)

FEI-NOVA NANOSEM-450 SEM was used to study the morphology of as-received raw materials. Specimens were subsequently polished for optical microscopy using SiC polishing papers and colloidal alumina (0.05µm) for wheel polishing. Keller's reagent was used as an etchant. Optical microscopy was done using LEICA image analyzer. Tecnai G2 20(FEI) S-Twin TEM was used to study the grains size and interface in the nanocomposites.

The phases and crystallite size in the powdered samples were analyzed using Panalytical X Pert Pro X-ray diffraction unit with Cu Kα radiation (kα=0.1542nm). XRD was done at step size of 0.03 with scan rate of 0.6 times/scan. Tensile testing of the specimens was done using Tinus Olsen H25KL-0030 tensometer. The hardness of the specimens was measured using Brinell Hardness Tester having steel ball (10mm) as indentor.

Results and Discussion

Figure 2 (a-c) shows the XRD patterns of all powder samples. The ratio of the peak intensity for the primary peaks of 6061Al powder has decreased after milling indicating entrapment of Al_2O_3 within the 6061 Al matrix due to mechanical alloying. The peak width has increased after mechanical alloying indicating decrease in crystallite size. The shifting of the main peak to higher 2θ angle indicates the existence of strain in the MA 6061 Al matrix powder. Infact, the line broadening of the main peak takes place due to both lattice strain and grain size reduction caused by MA [15-16], and same is in the present case.

As in Fig 2(b), none of the Al_2O_3 peaks are visible. This is due to nano size of alumina particles in the matrix. Due to nanosize of Al_2O_3 particles, sufficient numbers of lattice planes are not available, due to which intensity of diffracted peaks remains un measurable. In many cases when nanometric phases exist in a structure, they couldn't be investigated well by XRD, as reported by many researchers as well [17-18].

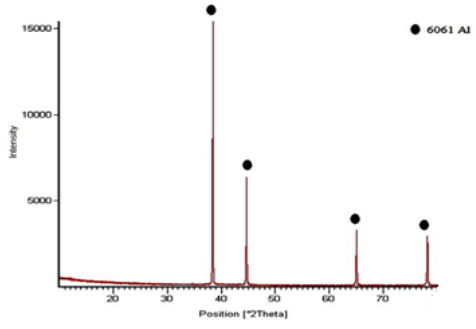
The crystallite sizes in the powders, as calculated by using following Scherer relationship [19], are shown in the Table3.

$$L = \frac{.94\lambda}{B \times \cos\theta} \quad (1)$$

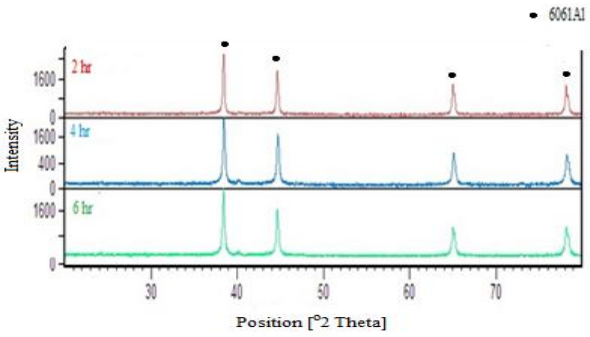
Where; B is peak width, λ = 0.154 nm and L is crystallite size.

Table 3: Crystallite size in different powders

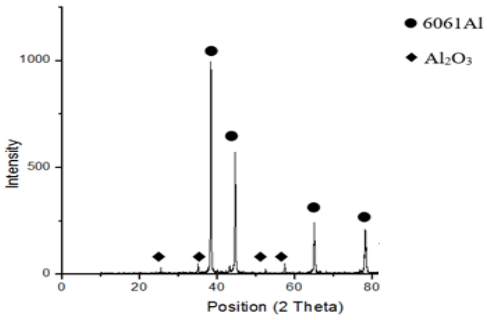
Powder sample	Crystallite size
Sample 1	107nm
Sample 4	45nm
Sample 5	56nm



(a)



(b)



(c)

Figure 2: XRD patterns of: (a) Sample 1; (b) Sample 2,3,4; (c) Sample 5

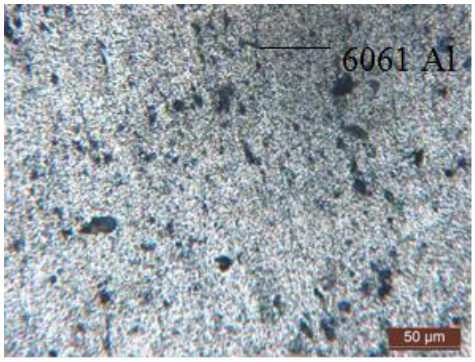


Figure 3: Optical micrograph of hot pressed 6061Al (specimen 1)

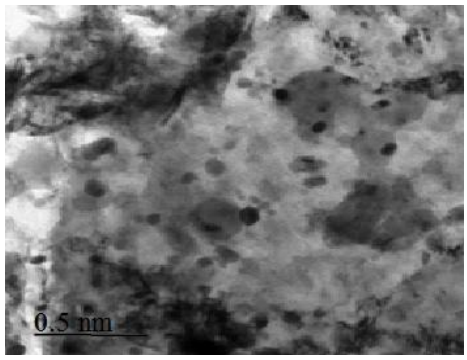


Figure 4: TEM micrograph for specimen 2

Optical micrograph of hot pressed 6061Al powder specimen (Fig. 3) shows precipitates which were formed during the hot pressing. TEM micrograph of specimen 2 is shown in Fig. 4. It can be clearly observed that the distribution of precipitated particles and dispersoid is uniform throughout the matrix. A uniform distribution of micro size Al_2O_3 particle in specimen 5 can be seen in its optical micrograph as shown in Fig.5.

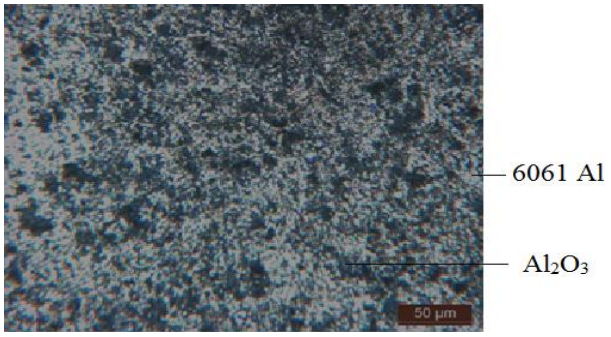


Figure 5: Optical micrograph of specimen 5

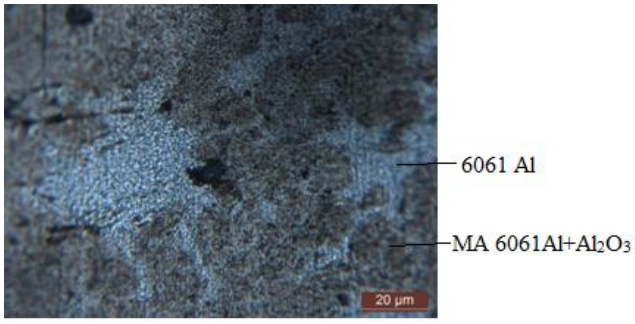


Figure 6: Optical micrograph of specimen 6

Figure 6 shows optical micrograph of bimodal composite (specimen 6) , where the white region is 6061Al powder and dark region is MA 6061Al matrix along with dispersed Al_2O_3 nanoparticles. Agglomeration of Al_2O_3 particles, as visible in Fig. 6, is due to the Y-cone mixed part of the specimen and it certainly deteriorates mechanical properties of the bimodal composite.

TEM micrograph of specimen 6, as shown in Fig.7, shows average grain size of about 1µm while the area marked as square shows ripples of ductile flow in hot pressed 6061Al powder, while the circle shows brittle (devoid of the ripples) region. Thus, the matrix of the bimodal nanocomposite is consisting of ductile as well as brittle regions. Fig. 8 (a) shows a clean Al_2O_3-matrix interface showing minimum interface reaction (It is interesting to see the lattice planes of Al_2O_3 particles in Fig 8(b)).

Mechanical Behavior

Ultimate tensile strength, % elongation and hardness obtained for the composite specimens are shown in Table 4.

Table 4: Ultimate tensile strength, % elongation and hardness of specimens

Specimen no.	UTS (MPa)	Elongation%	Hardness (HB)
1	115.9	18.5 %	36.2
Nanocomposite			
2	137.6	8.2 %	71.6
3	125.6	8.0 %	85.7
4	103.8	6.3 %	125.3
Microcomposite			
5	152.4	7.3 %	46.9
Bimodal composite			
6	148.3	8.7 %	74.6

Nanocomposite

Figure 9 shows the plot of tensile strength with MA duration of 6061Al - 10wt% Al_2O_3 (50nm) specimens. It can be seen that tensile strength is increased (by 19%) in

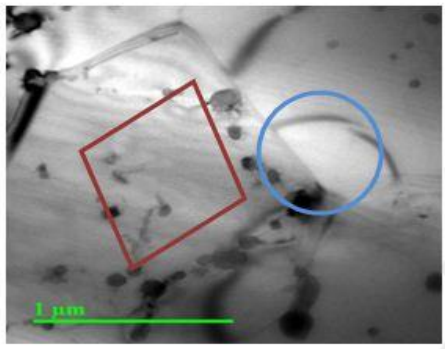


Figure 7: TEM micrograph of specimen 6

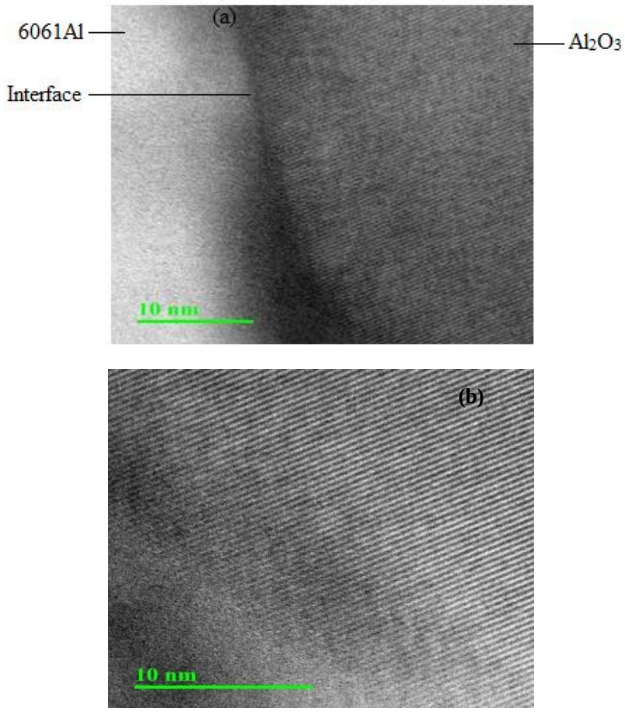


Figure 8: HR TEM image of specimen 6: (a) showing Al_2O_3-matrix interface; (b) lattice planes in Al_2O_3

2h mechanically alloyed specimen, as compared to unmechanically alloyed which can be attributed to dispersion strengthening. TEM micrographs of specimen 1 and specimen 2 are shown in Fig. 10 & 4 respectively. It can be seen that matrix grain size is of micron size (over one micron) in the unmechanically alloyed sample, which gets reduced to the sub-microscopic range on mechanically alloying for 2h. When MA duration is further increased to 4 h and 6 h, the tensile strength of nanocomposite is decreased due to reverse Hall-Petch effect when matrix grain size reduces to the nanometric range [19]. Due to nanograin size in the matrix, the matrix becomes unsuitable for further deformation as nanograins are considered to have minimum lattice defects (TEM micrographs of the specimen 3 and 4 could not be possible as they were very brittle).

Figure 11 shows the increase in hardness with MA duration for 6061Al - 10wt% Al_2O_3 (50nm). Hardness has increased almost by ~350 %. This increase in hardness is an attribute of the presence of harder nano Al_2O_3 particles in the nanocomposite, smaller means free path between neighboring nanoparticles, the greater constraint provided

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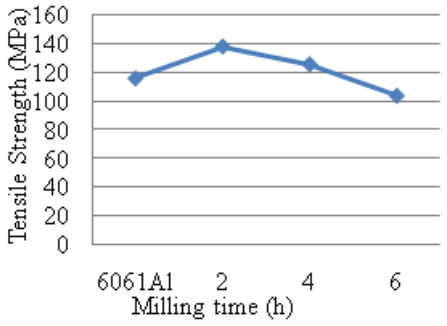

Figure 9: Tensile strength vs milling duration for 6061Al- 10wt% Al_2O_3 (50nm)

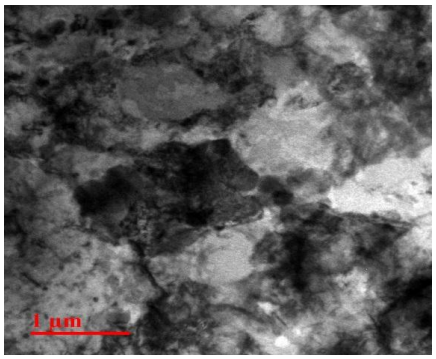

Figure 10: TEM micrograph for specimen 1

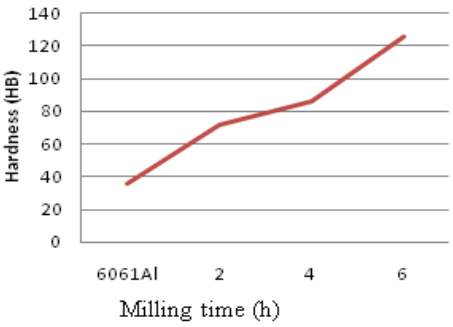

Figure 11: Hardness vs milling duration for 6061Al- 10wt% Al_2O_3 (50nm)

by the higher surface area of nanoparticles and decrease in grain size of the matrix [19].

Microcomposite

On addition of Al_2O_3 microparticles (10µm) in 6061Al matrix, the improvement in tensile strength (31%) is higher than nanoparticles addition due to dispersion strengthening effect provided by few nano Al_2O_3 particles present along with the micro Al_2O_3 particles, but improvement in hardness is incremental due to the lack of the beneficial factors available with nano Al_2O_3 particles as explained above.

Bimodal

Results for bimodal composite (Specimen 6) show improved tensile strength (28%) and ductility along with 200% improvement in hardness. This increase in tensile strength and ductility is due to the presence of ductile 6061Al matrix along with the work hardened (due to severe mechanical working) MA 6061Al matrix. Thus this bi-mode composite has optimal improvement in the tensile strength, ductility and hardness.

Conclusions

Mechanical alloying provides a uniform distribution of alumina particles in 6061Al matrix. Hardness in this composite can be greatly improved by incorporating Al_2O_3 nanoparticles while tensile strength can be improved by moderately mechanical alloying or by the addition of an optimal amount of Al_2O_3 nano particles. For optimal combination of hardness and tensile strength in the composite, bimodal or trimodal approach can be adopted. In general, selection of the matrix, reinforcement particles size, and the mechanical alloying duration should be chosen carefully keeping in view of the desired property improvement.

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