Development and implementation of a technology for introducing reinforcement into aluminum matrix composite

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Abstract. In this study, a new method for introducing reinforcement was developed and used in stir casting to fabricate aluminum matrix composites. Different reinforcements (microparticle SiC and nanofiber Al2O3) were incorporated into the technical aluminum AD0 (1050 ISO) aluminum alloy by a mechanical stirrer and then cylindrical specimens were cast. The microstructural characterization of the composite samples showed grain refinement of aluminum matrix with the introduction of reinforcement. The effects of reinforcement on the mechanical properties and impact behavior of the composites were investigated. Based on experiments, it was revealed that the presence of Al2O3 nanofiber reinforcement led to improvement in hardness. This was due to introduction of reinforcement and grain refinement of aluminum matrix. Studies on the fracture surfaces revealed that AMC with reinforcement is brittle, but in the case of nanofibers fillers, tough component was also present, which eliminated the risk of brittle fracture.

1. Introduction
Aluminum matrix composites (AMCs) offer high specific strength, high specific stiffness and hardness, good wear resistance, low thermal expansion coefficient. AMCs are successfully used in the aerospace, automotive, thermal management and bio-medical industries [1-7]. Normally the size of reinforcements generally ranges from a few nanometers to several hundred micrometers. Recently it is of interest to use nano-sized reinforcements to improve the properties of AMCs.

Various of methods for producing AMCs reinforced with nano-sized Al2O3 have been developed, including powder metallurgy [8, 9], spark plasma sintering [10], in-situ routes [11], stir casting [20]. For mechanical alloying, it normally involves mechanical mixing of Al powders and nano-Al2O3 for fabrication of bulk. Although the powder metallurgy techniques are more effective than the casting methods in terms of reinforcement distribution, uniform mixing of nano-sized reinforcements is lengthy, expensive, and energy consuming [12]. Furthermore, solid techniques seriously suffer from limitations in size and complexity of the components.

Melt processing which involves the stirring of nano-Al2O3 into Al melts, has some important advantages such as better matrix–reinforcement bonding, easier control of matrix structure, simplicity, low cost of processing, and nearer net shape and the wide selection of materials [12,13]. However, it is extremely difficult for the mechanical stirring method to distribute and disperse nano-sized reinforcements uniformly in Al melts due to high viscosity, low wettability in Al melts and large surface-to-volume ratio of nano-sized reinforcements. Furthermore, when the nano-Al2O3 is wetted in the aluminum melt, it will tend to sink to the melt due to the density difference between Al2O3 and aluminum. So nano-Al2O3 is hard to introduce into aluminum melt and easily agglomerated and clustered.
Regarding the mentioned challenges, introducing nano-Al₂O₃ and achieving to uniform dispersion is a principal purpose in production process of these composites.

The method combined with ball milling and stir casting is developed to fabricating Al-nano Al₂O₃ metal matrix composites. Different metallic particles are mixed with nano-Al₂O₃ as the reinforcements. Mazahery A. et al. [12] used the mixture of nano-Al₂O₃ particles (50 nm) and aluminium particles (16μm) as the reinforcements. Akbari et al. separately milled nano-Al₂O₃ particles with aluminum and copper powders and incorporated the mixture into A356 alloy using vortex method [13-16]. Copper acts as strengthening factor and, also affected particle dispersion [17].

In this research, different fillers were introduced into the Al melt, i.e., untreated SiC, milled particles Cu, and milled nanofiber Al₂O₃ - Cu composite powder.

2. Experimental procedure

2.1. Materials

In the current research, technical aluminum AD0 (1050 ISO) was chosen as the matrix alloy. The chemical composition of AD0 according to GOST 4784-97 is shown in table 1. Table 2 shows the properties of technical aluminum AD0.

| Composition | Al | Fe | Si | Zn | Cu | Mg | Ti | Mn |
|-------------|----|----|----|----|----|----|----|----|
| wt.%        | >99.5 | <0.4 | <0.25 | <0.07 | <0.05 | <0.05 | <0.05 | <0.02 |

Table 2. Properties of cast technical aluminum AD0.

| Material | Density (g/cm³) | Brinell Hardness (MPa) | Young’s modulus (GPa) | Tensile strength (MPa) |
|----------|----------------|------------------------|-----------------------|-----------------------|
| AD0      | 2.71           | 200                    | 71                    | 60                    |

Discontinuous nanosized alumina fibers with an average diameter of 10-20 nm (ANF Technology Ltd, Estonia, table 3) and micro copper powders with average size of 20 μm were used as components of reinforcement powders. The powders were mixed in the mass ratio of Cu/Al₂O₃ = 1. Mixed powders were milled in a mortar for 20 min.

Table 3. Properties of Al₂O₃ nanofibers.

| Material       | Density (g/cm³) | Thermal resistance (°C) | Young’s modulus (GPa) | Tensile strength (GPa) |
|----------------|----------------|-------------------------|-----------------------|------------------------|
| Al₂O₃-nanofibers | 3.98           | 1200                    | 400                   | 12                     |

For comparative analysis, unreinforced alunium AD0 and composite reinforced with micro powders Cu, composite reinforced with microparticle SiC (17μm) were fabricated (table 4).

Table 4. Composition of the experimental samples.

| Specimens | Composition of the samples (wt.%) | Matrix | Reinforcement      |
|-----------|----------------------------------|--------|--------------------|
| 1         | AD0                               | -      |                    |
| 2         | AD0                               | 1%SiC (17μm) |                |
| 3         | AD0                               | 1%Cu (20μm) |                |
| 4         | AD0                               | 1%Cu (20μm) + 1%Al₂O₃ nanofibers |    |

2.2. Fabrication of composites

The composites were produced by stir casting method. Technical aluminum AD0 was charged into the ceramic crucible and heated up to 800°C (above the alloy liquidus temperature) for melting. The graphite stirrer fixed on the mandrel of the drilling machine was introduced into the melt. Reinforcements were introduced into the melt at a temperature of 800°C. Pure argon was used as the carrier gas for injection
of the reinforcements. After completion of the injection, the slurry was stirred at a rate of 600 rpm for 10 min. The composite slurry was poured into cast iron mold and shaped in the form of cylinder of 20 mm outer diameter and height of 80 mm. Figure 1 shows the schematic of the experimental set-up used in the production of the composites and the as-cast specimens.

![Figure 1](image)

Figure 1. (a) Schematic of the experimental set-up used in production of the composites. (b) As-cast specimens.

2.3. Composite characterizations
The morphology of the Cu powder, nanofiber Al₂O₃ and the Cu-nanofiber composite powders were observed using a optical microscope (Olympus GX51).

To study the microstructure, samples were cut and prepared by grinding through 800 and 1200 grit papers followed by polishing with 9,3,1μm diamond paste, and etched with Keller’s reagent (2 ml HF (48%), 3 ml HCl (conc.), 5 ml HNO₃ (conc.) and 190 ml water). Microscopic examinations of the composites and matrix alloy were characterized by optical microscope (Olympus GX51).

To study the microhardness, the Vickers microhardness values of the samples were measured on the polished samples using an DURASCAN 70 microhardness tester with a load of 100 g. For each material, 100 points on an area of 100 mm² were tested in order to study the frequency distribution of microhardness values.

Charpy impact test specimens pre-crack notches were machined from each casting according to GOST 9454-78. Fractographic examinations were also carried out on the fracture surfaces of the broken specimens by means of Tescan Vega II LMH scanning electron microscope (SEM).

3. Results and discussion
3.1. Powder characterization
Figure. 2 shows the morphology of as-received Cu powders and nanofibers Al₂O₃, milled Cu powders and milled nanofiber Al₂O₃–Cu composite powders. After milling, most of the Cu powders become smaller. Because of the Van der Waals force nanofibers Al₂O₃ are easily agglomerated (figure. 2b). In addition, these aggregates may have entrapped air, which could lead to difficulty in their incorporation
into liquid Al melt, as well as result in the incorporation of gas bubbles within the microstructure of the composite as the aggregates are drawn beneath the surface of the melt [10]. Also, these nanofibers Al₂O₃ have poor wettability with melt [18,21]. Figure 2d reveals that nanofibers Al₂O₃ have covered the surface of Cu powders. In addition, due to the milling effect, the nanofibers are not agglomerated and the distribution is more uniform. Furthermore, milling remove air on alumina nanofibers, free oxygen on the surface of alumina nanofibers reacts with Cu, leading to the formation of CuO/Cu₂O. When composite powders are incorporated into the molten alloy, CuO/Cu₂O will react with Al melt, leading to the formation of new Al₂O₃.

Figure 2. Image of as-received Cu powders (a), nanofibers Al₂O₃(b), milled Cu powders (c) and milled nanofiber Al₂O₃–Cu composite powders (d). 100x

3.2. Comparative analysis of the microstructure
The microstructural analysis was performed on the technical aluminum AD0, AD0+1%SiC, AD0+1%Cu, AD0+1%Cu+1%Al₂O₃. As shown in figure 3, after the introduction of SiC microparticles or Cu, the structure is refined. When Al₂O₃ nanofibers are added, decreasing is more efficient.

Figure 3. Microstructure of experimental samples:
(a) AD0; (b) AD0+1%SiC; (c) AD0+1%Cu; (d) AD0+1%Cu+1%Al₂O₃ (200x)

3.3. Microhardness
Figure 4 displays the frequency distribution of microhardness values of the specimens. In figure 4 (a) the frequency distribution of microhardness values of technical aluminum AD0 is approximately uniform and symmetric. After introducing 1% micro-sized SiC particles (Figure 4 (b)), the hardness is increased but the frequency distribution symmetry is destroyed, which is because composite materials contain a soft matrix and hard reinforcement phase. When introducing 1% Cu, the hardness distribution is wider (Figure 4 (c)). After introducing 1% Cu (20μm) + 1% Al₂O₃ nanofibers into the Al melt (Figure 4 (d)), the hardness is increased 18% . The frequency distribution obviously consists of two parts: soft matrix part and reinforced part. The dispersion of Al₂O₃ nanofibers enhances the hardness, as Al₂O₃ is harder than aluminum matrix. And the higher hardness of the composites could be attributed to the fact the Al₂O₃ nanofibers act as obstacles to the motion of dislocation. The hardness increment can also be attributed to reduced grain size.
Figure 4. Histogram of distribution of Vickers microhardness values: (a) AD0; (b) AD0+1%SiC; (c) AD0+1%Cu; (d) AD0+1%Cu+1%Al2O3

3.4. Impact behavior
As depicted in table 5, the absorbed impact energy of aluminum matrix composites with 1% SiC, 1% Cu and 1%Cu+1%Al2O3 is found to be lower compared to technical aluminum AD0. The reduction in impact energy can be explained by the fact that a stress concentration may exist near the reinforcements. In addition, the agglomeration of the reinforcements is easily separated under impact loading. Similar findings have been observed in other studies of aluminum matrix composite [19]. Compared with the sample of AD0+1%Cu, the absorbed impact energy of aluminum matrix composites with Al2O3 is increased. The introduction of Al2O3 fibers has a positive effect on impact behavior.

The fracture surface morphologies of the samples were shown in figure 5. Elongated dimples were visible on the fracture surface of the sample of matrix composition AD0. A rough surface indicates that the matrix alloy exhibits serious plastic deformation before fracture. The presence of hard reinforcements SiC17 and Al2O3 embrittle the material, which is expressed by the nature of the development of the crack. The AD0 + 1% Cu sample has a relatively smooth fracture surface, and there are a large number of tiny dimples on the surface. Compared to the sample, after the introduction of Al2O3, the nature of the crack propagation changes, but the size of a single dimple increases, which indicates the presence of a toughness component of the fracture. The results of fractographic studies are consistent with the data of mechanical properties under impact.

| Materials          | Absorbed impact energy (J) |
|--------------------|-----------------------------|
| AD0                | 67.4±5.9                    |
| AD0+1%SiC          | 34.9±9                      |
| AD0+1%Cu           | 22.5±2.2                    |
| AD0+1%Cu+1%Al2O3  | 31.3±1.1                    |
Figure 5. Fracture surface morphologies of (a) AD0, (b) AD0+1% SiC, (c) AD0+1% Cu, (d) AD0+1% Cu+1% Al$_2$O$_3$ (300x)

4. Conclusion
In general, Al$_2$O$_3$ nanofibers are incorporated into aluminum matrix with the help of micro-Cu particles mixing and under the processing conditions of stir casting used in this study.

Based on the present study, the following conclusions can be drawn:

1. As a result of the work, samples of a composite material with alumina fibers with a diameter of 10-20 nm are obtained.
2. In the course of the study, the effect of introduction of reinforcements on structure refinement is established.
3. The general patterns of changes in hardness with a change in component composition are established.
4. Analysis of impact behavior show that AMC with hard reinforcement is brittle, but in the case of nanofibers fillers, tough component is also present, which eliminates the risk of brittle fracture.

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