Effect of Al₂O₃ Coated Cu Nanoparticles on Properties of Al / Al₂O₃ Composites

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Abstract
Aluminum matrix composites reinforced with various contents of Al₂O₃ nanoparticles coated with Cu (0, 5, 10 and 15 wt. %) were prepared by powder metallurgy technique. Al₂O₃ particles are coated with 30 wt. % Cu by electroless deposition after surface activation using 10 wt. % silver. Appropriate amounts of Al and Al₂O₃ (coated with Cu) are well mixed in a ball mill (4:1 balls to powder ratio) for 6 h. Then, the mixture is sintered by hot pressing at 500°C under 700 MPa uniaxial pressure for 45 min in an argon atmosphere. Phase identification and microstructure of sintered samples are studied. The density, thermal expansion and mechanical properties were measured. Increasing Al₂O₃ nanoparticles coated with Cu highly influences composite samples. Good distribution of Al₂O₃ coated with Cu in the Al matrix and their improved wettablility, improve the microstructure, hardness, compressive strength and thermal expansion properties. However, addition of Al₂O₃ coated with Cu shows a deteriorated effect on both densification and ductility.

Keywords
Nanocomposite- Electroless deposition-Al composite - Mechanical properties - Microstructure

Introduction
Recently, metal matrix nanocomposites (MMNCs) with uniform dispersion of particles smaller than 100 nm size have attracted attention due to their excellent mechanical, tribological, electrical and thermal properties compared with metal matrix composites (MMC) [1,2]. Moreover, many studies indicated that adding a small fraction of nanoscale reinforcement significantly improves mechanical and physical properties; as elasticity modulus, fatigue, specific stiffness, wear resistance, creep rate, thermal and electrical conductivity, coefficient of thermal expansion, thermal stability, etc. [3,9].

Microstructure and mechanical attributes of nanocomposites and microcomposites are burning areas of research [10,11]. Aluminum matrix composites (AMCs) play a crucial role in various fields, as automotive, ground transportation and aerospace industries [12]. It is also used in electronics and energy sectors for its highest electrical and thermal properties and good mechanical properties [13, 14]. During the last decade, researches on the fabrication of Al-based composites using various nanoparticulate reinforcing phases in micro and nanoscale were conducted. Examples are h-BN [15], Carbon Nano Tubes (CNTs) [16], TiC[17], graphite[18], Al₂O₃[14], TiB₂[14], Fe₂O₃ [20], CuO[21], SiO₂ [22], B₄C[23], AlN [24], NiAl₃ [25] and Graphene [26]. Compared with such reinforcements, alumina particulates highly improve the mechanical and tribological properties. Therefore, alumina particulates are widely used in AMCs [3]. Al–Al₂O₃ particulate composites possess the properties of Al, as excellent thermal and electrical conductivities, high formability, and light weight. Such composites possess also properties of Alumina, i.e. low thermal expansion coefficient and high mechanical properties [14, 27]. They are widely used in automotive, aircraft, aerospace, and military industries, and as electrical contacts and bearing materials in many applications, due to their good thermal and electrical conductivities, and their high strength/weight ratio [24-28].

Al–Al₂O₃ composites are prepared and investigated by several groups [29]. Mosawi.B.T Al-Al₂O₃ nano-composites was prepared by ball milling and uniaxial hot compaction [29]. Also, Al–Al₂O₃-Mg nanocomposite was prepared by mechanical alloying, and it was found that the addition of Mg improved the mechanical and thermoelectrically properties [30]. Al–Al₂O₃ composite was synthesized by spark plasma sintering (SPS) [11], and by injecting reinforcing particles into molten Al alloy [30]. It was concluded
reported that the ultimate tensile strength and hardness of Al-2024-\(\text{Al}_2\text{O}_3\) increases with increasing alumina content [31]. Alumina particles act as barriers to dislocations, and the increase of its content increases hardness, compressive strength and Young’s modulus, however decreases ductility. Furthermore, it was noticed that sintering Al-\(\text{Al}_2\text{O}_3\) composites by microwaves enhances their performance [30]. Besides, mixing Al and \(\text{Al}_2\text{O}_3\) powders without coating results usually in weak wettability and bonding between both phases [31]. Moreover, coating \(\text{Al}_2\text{O}_3\) by Ni leads to superior properties [32].

The addition of \(\text{Al}_2\text{O}_3\) to Al matrix improves mechanical properties, but it reduces thermal and electrical properties as well as ductility [33]. The effect of coating \(\text{Al}_2\text{O}_3\) with Cu on Al-\(\text{Al}_2\text{O}_3\) composite properties is not reported yet. Hence, present work deals with the production of Al-\(\text{Al}_2\text{O}_3\) composites by powder metallurgy and hot compaction. Coating of alumina with Cu is carried out by electroless deposition before adding to the matrix. The coating process leads to get more homogenous reinforcement, excellent surface bonding, improved wettability, pure interphase, and appears to be a suitable method for preparing (MMNCS).

**Experimental Work**

Commercial pure Al powder with purity 99.99% (Dop organic kimya, Ankara, Turkey) and average particle size < 10 µm is used as a matrix. The applied reinforcement is alumina powder with purity 99.99% and 40 nm average particle size (M K Impex CORP. Canada.). Silver nitrate, copper sulfate, sodium hydroxide, potassium sodium tartrate (rochelle salt), ammonia and formaldehyde are used for electroless coating of silver and copper on \(\text{Al}_2\text{O}_3\) nanoparticles. The manufacturing of Al matrix reinforced with \(\text{Al}_2\text{O}_3\) coated with Cu nanocomposites were processed in four stages as shown in Fig. 1.

The electroless plating does not depend on the electrical current as the electroplating, but depends on the charge transfer that takes place through chemical reduction. Copper precipitation in electroless plating process takes place by catalytic reduction using a reducing agent. \(\text{Al}_2\text{O}_3\) is the core of the electroless plating process. Because \(\text{Al}_2\text{O}_3\) particle surface is nonconductive, the coating process of \(\text{Al}_2\text{O}_3\) with Cu is difficult to take place directly. Therefore it is necessary to make pretreatment of the \(\text{Al}_2\text{O}_3\) surface. The impurities on the surface should be eliminated using the sensitization process by immersing in 10 wt.% sodium hydroxide solution and stirring for 1hr. The powder is then immersed in acetone and stirred with a magnetic stirrer for 1hr. It is finally filtrated and washed with distilled water, then dried in oven at 110° C for 1h. The activation process of \(\text{Al}_2\text{O}_3\) is applied by deposition of 10wt. % silver on the surface of \(\text{Al}_2\text{O}_3\) nanoparticles. Hence, 3g/l silver nitrate, 300 ml formaldehyde and ammonia so that the PH is from (11-13) are added to the chemical bath. There upon, it is possible to make electroless deposition of copper on surface of \(\text{Al}_2\text{O}_3\) nanoparticles. Copper coating takes place using copper sulfate pentahydrate (CuSO4.5H2O), formaldehyde (HCHO) and Rochelle salt (C4H4O6KNa.4H2O) (35gm, 200ml and 170gm), respectively for each liter. Sodium hydroxide (NaOH) is add to adjust PH value > 11. Copper sulfate is the source of copper that is insoluble at high PH values, therefore Rochelle salt is added to make it soluble. Formaldehyde is used as a reducing agent, and it is addition donates the electrons to Cu, so that it can coat the \(\text{Al}_2\text{O}_3\) nanoparticles. After the activation and coating process the powder is washed three times by distilled water and dried at 110° C for 1h. Then the copper on the powder surface may oxidize. Therefore, it is reduced by hydrogen in tube furnace at 500° C for 2hr.

![Figure 1 Schematic diagram of the sequence of Al/Al2O3 coated with Cu composites fabrication.](image-url)
The weight of Cu coating is determined by the equation
\[ \Delta W = W_2 - W_1 \]  \hspace{1cm} \text{Eq. 1} 

Where \( W_2 \) and \( W_1 \) represent mass of \( \text{Al}_2\text{O}_3 \) after and before coating, respectively.

\( \text{Al} \) and mixtures of \( \text{Al} \) and 5, 10 and 15 wt% \( \text{Al}_2\text{O}_3 \) coated powders were performed by a mechanical mixer in a ball milling machine using 4:1 ball to powder mass ratio (BPR) for 6 hrs. The mixing procedure was carried out at room temperature and the rotation speed of the ball mill was fixed at 300 rpm for 6 hrs to achieve a homogenous distribution of powder mixture, the mixing vial was filled with argon gas before mixing as protective atmosphere to avoid any influence of oxidation during mixing process. Additionally, to avoid excessive temperature in the mixing vial the ball mill machine was given 20 min set after every 40 min of the ball mixing duration. Then the mixtures were consolidated by hot pressed at sintered temperature 500°C with applied heating rate 10°C/min under high uniaxial pressure 700 MPa under an argon atmosphere to prevent oxidation of powders during sintering process for 45 min dwell time on the 12 mm internal diameter die.

The main phases in as received powder and \( \text{Al}_2\text{O}_3 \) coated with Cu and also the composition after consolidation are emphasized by X-Ray diffraction (model 5000) operated at 40 kV and 30 mA with Cu K radiation source (10⁰ to 10⁰ scanning 2θ range at step size 0.05°).

Sintered nanocomposite samples are ground by 500-2500 grit papers, then polished by FE-SEM connected with energy dispersive X-ray (EDX) spectrometer to check the distribution of constituent elements. The crystallite size of sintered samples is calculated by Scherrer equation.

The densification parameters of consolidated samples is measured in aqueous media according to Archimedes principle, using deionized water as a floating liquid and a balance of 0.1 mg accuracy.

Thermal strain of composites is measured using a digital indicator with 0.001 mm accuracy and electrical furnace; Fig.2.It is measured at temperatures in the range of 150-450 °C for 10 min. Each measured value is repeated 3 times. The thermal expansion value at each fixed temperatures is determined from the following equation
\[ \alpha = \frac{\Delta l}{l_0 \Delta T} \]  \hspace{1cm} \text{Eq. 2} 

Macrohardness of the specimens is measured using a Vickers hardness tester at room temperature, on flat and smooth surfaces. Each value is an average of at least 6 random indentations under 300 gf for 15 s.

Compression strength of investigated samples is measured using uniaxial 350 KN capacity "Instron" machine. The cylindrical test samples are 113 mm² cross-sectional area and 1:1 aspect ratio. Grease is used to minimize friction between the sample and the compression machine. The applied cross-head speed of the universal test machine is 0.5 mm/min and the test is conducted at room temperature. To ensure the accuracy of test three specimens will tested for each category.

### RESULTS AND DISCUSSION

The electroless plating of \( \text{Al}_2\text{O}_3 \) ceramic by metal lead to improve the wettability between reinforcement and matrix in order to enhance microstructure and mechanical properties. Fig.3a and Fig.3b show FE-SEM micrographs microstructures of the used \( \text{Al} \), \( \text{Al}_2\text{O}_3 \) powders, respectively. It indicates that \( \text{Al} \) and \( \text{Al}_2\text{O}_3 \) powders have spherical and irregular shapes, respectively. Fig.3c shows the microstructure of \( \text{Al}_2\text{O}_3 \) nanoparticles after Cu electroless coating. It indicates good distribution of Cu particles on the surface of \( \text{Al}_2\text{O}_3 \). This reveals successful coating with Cu. Fig.3d of the EDX spectrum analysis of coated \( \text{Al}_2\text{O}_3 \) shows the presence of Cu, Ag and \( \text{Al}_2\text{O}_3 \) without any unwanted elements formation during electroless plating process. The good coating of \( \text{Al}_2\text{O}_3 \) particles reduces the agglomeration and increases the homogenous distribution in \( \text{Al} \) matrix during mixing as shown in Fig.3e[2,4].

Figur.4 shows the XRD pattern of coated \( \text{Al}_2\text{O}_3 \) nanoparticles with Ag and Cu. All diffraction peaks in pattern are compared with the reference card to check the presence of different phases. Only three phases are already identified, which also confirms the successful electroless plating of \( \text{Al}_2\text{O}_3 \) particles without impurities.

The XRD analysis is shown in Fig.5. \( \text{Al}_2\text{O}_3 \) coated with copper is shown in samples containing several alumina contents in \( \text{Al} \); i.e., prepared \( \text{Al}-\text{Al}_2\text{O}_3 \) nanocomposite. No new peaks appear in Fig.5 indicating that, no new phase is formed by reaction between \( \text{Al} \) and \( \text{Cu} \) during sintering. Also, slight peak broadening can be seen with increasing the content of coated \( \text{Al}_2\text{O}_3 \) from 0, 5, 10 and 15 wt.%, that attends the decrease of crystallite size 250 ,158.2, 92.9 and 78.3, respectively. For low volume fractions of \( \text{Al}_2\text{O}_3 \) the intensity of peaks is very low as compared to \( \text{Al} \) matrix. Additionally, the amounts of Cu and Ag are not detected by XRD, but are detected using EDAX.

Figur.6 shows FE-SEM of samples after consolidation at 500°C. The samples contain different contents of coated \( \text{Al}_2\text{O}_3 \), (0, 5, 10 and 15 wt.%). All
micrographs show a homogenous distribution of Al2O3 coated with copper. The homogenous distribution is due to wettability and low surface energy of coated Al2O3 and Al matrix [12] that decrease micro-voids and enhance mechanical properties. To confirm the main constituents of the composite using X-ray and EDS analysis as shown in Fig.7. Fig.7a indicates the uniform distribution of constituents without agglomeration. EDS in Fig.7b shows the main constituents (Al, O, Ag and Cu) of composite and their atomic percent, without any other constituent during sintering process.

Figure 3 Microstructure of Al/Al2O3 nanoparticles (a) Al as received (b) Al2O3 before coating, (c) Al2O3 after coating, (d) EDX of powder after coating and (e) Al/Al2O3 coated with Cu nanoparticles after mixing

Figure 4 XRD of Al2O3 nanoparticles after Ag and Cu coating.

Figure 5 XRD analysis of Al-Al2O3 coated Cu nanocomposite after consolidation.
The effect of Cu-coated Al₂O₃ nanoparticles as reinforcement on relative density of Al matrix is shown in Fig.8. The relative density of sintered samples containing 0 wt. % Al₂O₃ is close to 100%. The relative density increases at its sample due to hot compaction method with high pressure and temperature applied. Another reason, the fine particles size of Al powders act as increases the surface area and diffusion, and leads to a final density close to theoretical value. Increasing the content of alumina coated with Cu from 0, 5, 10 and to 15wt.% leads to decrease the relative density of composite (porosity increases from 0.43 to 5.68%) compared to the theoretical density. This is primarily attributed to the increase of Al₂O₃/ Al₂O₃ contact surface area between the nanoparticles of Cu-coated Al₂O₃ and aluminum that leads to poor bonding between matrix and reinforcement. The increased percent of Al₂O₃ hard particles resists deformation during pressing consequently increases grain boundary spacing lead to reduction of the densification. Additionally Al₂O₃ particles have irregular shape with sharp edges acting as entrapping air between particles resulting from that pores[4].

Vickers hardness values of Al/ Al₂O₃ (coated with Cu) nanocomposites at 300gm load for 15s are shown in Fig.9. The hardness of sintered samples increases gradually by increasing the Al₂O₃ content from 0 to 15 wt.%. It increases to 87.8 HV for samples containing 15 wt. % Al₂O₃ coated with Cu nanocomposite compared with 53.2 HV for pure Al, i.e 65% improvement. This improved hardness is attributed to the presence of second phase hard ceramic nanoparticles acting as load-supporting components, and as constraint to plastic deformation of the matrix during indentation that, leads to hardness increase. The metal Cu nanoparticles coated on Al₂O₃ enhances good wettability between Al₂O₃ and Al matrix. Hence, improving the uniform distribution of Al₂O₃ in the Al matrix, therefore the bonding between the Al matrix and the reinforcement is getting better, and acts as strong barrier for plastic deformation of the soft matrix. Moreover, the crystallite size is reduced to 78.3 nm at 15wt% Al₂O₃ coated with Cu. Hence, the grain boundary area increases that impedes the movement of dislocations, and subsequently the hardness increases.

**Figure 6** SEM micrographs of hot compacted Al- Al2O3 composites with various alumina contents (a) 0 wt.%, (b) 5 wt.%, (c) 10 wt.% and (d) 15 wt.%.
Figure 10 shows the effect of prepared nanocomposite content of on its compressive strength. It is obvious that, the compressive strength increases with increasing reinforcement content up to 15 wt. %. Because it is not possible to determine the compressive yield strength and the ultimate compressive strength, the mechanical behavior of samples is chosen via conditional compressive deformation resistance at 10% of compressive ratio.

At 10% compressive ratio, the samples Al2O3 contents are 0%, 5%, 10% and 15% and the loads are 163.69, 217.50, 253.53 and 336.5 MPa, respectively. The compressive strength increases up to 105 % for samples containing 15 wt. % Al2O3 coated Cu than that of Al pure samples. Composite strengthening could be due to several mechanisms, as dispersion hardening. Homogenously dispersed Al2O3 nanoparticles coated with copper along grain boundaries of aluminum act as obstacles of dislocation movement in the matrix[5]. Also, the grain size becomes finer with increasing the content of Al2O3 nanoparticles coated with copper, leading to more grain boundaries in the composite. Besides, coating Al2O3 with Cu enhances the wettability and hence the bonding of Al2O3 and Al matrix. This is responsible of load transfer from matrix to reinforcement that leads to the enhancement of compressive strength.

The measured CTE value is shown in Fig.11. The increase of CTE of samples containing 0 wt.% Al2O3, is due to the high CTE of Al under the thermal load. Also, the decrease of CTE with increasing Al2O3 content may be due to low CTE and homogeneously distributed Al2O3 particles in the Al-matrix as shown in Fig.6. Increasing Cu- Al2O3 wt. % and its good distribution in the matrix, good bonding and good wettability restricts the aluminum expansion especially at high temperatures especially higher than 300°C.

Conclusions

Based on present results the following can be concluded:

- The surface of Al2O3 nanoparticles can be activated and coated successfully with Ag and Cu by electroless deposition technique.
- Al/Al2O3 coated with Cu composite can be well prepared using powder metallurgy.
- 3) Reinforcing Al2O3 nanoparticles coated with Cu are homogenously distributed throughout the Al matrix. Also, the desired relative density of composite indicates that the applied technology may effectively fabricate Al-Cu coated Al2O3 nanocomposite.
- The relative density is reduced slightly by increasing Cu coated Al2O3 nanoparticles content, but the apparent porosity is increased, which can be attributed to the formation of micro pores during consolidation process.
- Hardness of Al increases from 53.2 to 87.8 HV by adding 15 wt. % Al2O3 coated with Cu.
- The compressive strength of the composite increases with increasing Al2O3 coated with Cu nanocomposite content. This is attributed to grain size reduction, and increase of dislocation density.
- Higher contents of Al2O3 coated with Cu reduces the expansion coefficient of nanocomposite.

Nomenclature

| MMNsCs | Metal matrix nanocomposites |
|--------|----------------------------|
| AMCs   | Aluminum matrix composites |
| FE-SEM | Field emission scanning electronic microscopy |
| EDX    | Energy dispersive X-ray |
| HV     | Hardness Vickers |
| BPR    | Balls to powder mass ratio |
| \( \alpha \) | Coefficient of thermal expansion |
| \( \Delta T \) | The temperature difference between the tested and room temperature. |
| \( \Delta L \) | Thermal strain |
| Lo     | Length of the sample |

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