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Microstructure and wear behavior of Al-Al_2O_3 *in situ* composites fabricated by the reaction of V_2O_5 particles in pure aluminum

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Abstract: In this study, fabrication of Al based *in situ* composites and their formation mechanism is investigated through the stir casting approach. *In situ* composites were prepared with the addition of various amounts of V_2O_5 particles in pure aluminum. Due to the reaction of vanadium pentoxide with the molten aluminum, Al_2O_3 (alumina) is formed. X-ray diffraction (XRD), scanning electron microscopy (SEM) and microscopy were used for the examination of the produced *in situ* composites. Increment in hardness was observed in cast *in situ* composites as compared to the pure aluminum. Friction and wear behaviors of cast *in situ* composites were studied by using a pin on disc tribo tester. The investigation showed that the wear loss and coefficient of friction increases with the load. The result indicates that wear rate increases with higher percentage of addition. The different phases formed are shown by the XRD study and the presence of Al_3V and alumina in the composite enhanced the properties.

Keywords: alumina; hardness; *in situ* composite; V_2O_5; wear.

1 Introduction

Worldwide, aluminum alloys has a wide range of applications in many areas due to the advantages of its associated mechanical and tribological properties [1–3]. Among nonferrous materials, aluminum shows better strength to weight ratio, resistance to corrosion, ductility and very low cost [4]. However, the applications of pure aluminum are restricted due to their poor wear resistance and softness. This problem is ended by the reinforcement of hard particles in aluminum and its alloys. A number of hard ceramic particles like SiC [5], TiB_2 [6], MoO_3 [7], ZnO [8], ZrB_2 [9], TiO_2 [10] etc. were incorporated in aluminum alloys to enhance their wear properties. Great improvement has been reported in wear and mechanical properties by the addition of nonmetallic phases like borides, nitrides, oxides and carbides into aluminum alloys [11, 12]. Reinforcement of particles in the aluminum matrix can be achieved by two methods; *ex situ* and *in situ*. The reinforcement particles can be externally added or can be generated within the matrix. Aluminum based *ex situ* composites show low bonding strength between the matrix and reinforcement, whereas *in situ* composites have better adhesion [13, 14]. The *in situ* method is very economical and provides better mechanical and wear properties as compared to *ex situ*, due to uniform distribution of reinforcement particles into matrix, and formation of thermodynamically stable phases [15].

A new technique, particulate reinforced metal matrix composite (PRMMC), has been suggested for the formation of Al_2O_3 particulates by the *in situ* reaction. The SO_3 decomposed from $Al_2(SO_4)_3$ improves the bonding between aluminum based matrix and Al_2O_3 and provides good strength [16]. Another work for the formation of alumina particles by the *in situ* reaction has been reported. In this case, alumina particles were formed by the reduction of CuO and silicon oxide. The tensile properties were significantly improved due to the presence of alumina particles. The strain hardening effect was observed due to the presence of particles in the composite sample [17]. A chemical reaction between CeO_2 and molten aluminum was carried out by the direct molten reaction (DMR) process for the formation of Al_2O_3 by the *in situ* reaction. The investigation reveals that the strength of cast composites was increased because of Al_2O_3 particles. High density dislocation around the alumina particles was found to be responsible for the composite strengthening [18]. A much less amount of energy is required to produce aluminum as compared to the steel. Aluminum composites are strong and infinitely recyclable. This has very favorable impact on the environment. None of its production processes involve significant risks for health

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or the environment. The *in situ* reaction takes place within the melt and only heat escaped out. In fact, the aluminum industry has been very concerned over the years in reducing pollution and the effects on the environment.

In the present work, an *in situ* approach has been used for the preparation of Al-Al_2O_3 composites by the reaction between pure aluminum and V_2O_5 particles. The intention of this study is to investigate the microstructure and wear properties of *in situ* aluminum composites obtained by the dispersion of V_2O_5 particles in different amounts. Addition of a small amount of magnesium improves the wettability of alumina particles and enhances the reaction process.

2 Materials and methods

2.1 Fabrication and characterization of *in situ* composites

Commercially available pure Al and extra pure AR grade V_2O_5 particles were used in this experiment. The average size of V_2O_5 particles was 159.9 nm, as measured by a Malvern particle size analyzer. About 1100 g of pure aluminum containing 99.68% Al, 0.14% Fe and 0.18% Si was used. The elemental composition was determined by the optical emission spectrometer (SPECTROMAXx, Ametek, Germany). The temperature range for the Al/V_2O_5 composite was determined by differential scanning calorimetry analysis. Figure 1 represents the differential scanning calorimetry analysis for the sample selected; exothermic and endothermic peaks were found. The first endothermic peak starts at 650°C, which corresponds to the melting of aluminum and further it proceeds towards the exothermic reaction between aluminum and vanadium pentoxide subsequent to the formation of Al_2O_3. The exothermic peak was observed at around 850°C as per reaction (2) shown below, which results in the formation of Al_3V:

$$3V_2O_5(\text{powder})+10\ \text{Al}(\text{powder})\rightarrow 6\text{V}(\text{active})+5\,Al_2O_3+\text{heat} \quad (1)$$

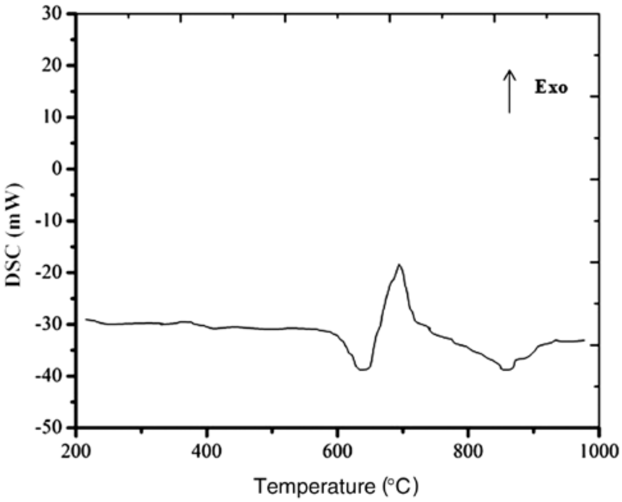

Figure 1: Differential scanning calorimetry (DSC) analysis of Al and V_2O_5.

Figure 2: Bottom pouring stir casting apparatus.

$$\text{V}(\text{active})+3\ \text{Al}(\text{molten})\rightarrow Al_3V(\textit{in situ}\ \text{in aluminium matrix}) \quad (2)$$

So, according to the reaction, the Al_3V intermetallic *in situ* compound is formed in molten aluminum. The aluminum is melted at 850°C in a bottom pouring stir casting furnace from Swam Equipment (Hasthinapuram, Chennai, Tamil Nadu, India) as shown in Figure 2. The stirrer blade used in the experiment is made of stainless steel and before use, the stirrer is coated with a fine paste of graphite and dried so that no dissolution of steel in molten aluminum takes place.

Experiments have been conducted with different percentages of vanadium pentoxide. The different amounts of V_2O_5 are weighed according to the particles selected for addition. Before the addition, particles are preheated at 250°C in order to remove moisture content or any other gases which are present in the reinforcement. Preheating of particles also avoids significant decrease in temperature [19]. The addition rate is controlled by the knob equipped in the setup. For melt temperature measurement, the thermocouple is placed in the melt chamber. The process parameters used in the experiment are mentioned in Table 1.

When the melt temperature reaches 850°C, a small amount (5 g) of Mg is added in the melt in the presence of argon gas and V_2O_5 is added

Table 1: Process parameters used.

Parameters	Variables used
Melt temperature	850°C
Particle preheating temperature	250°C
Die temperature	250°C
Stirrer rpm	800
Time of processing	15 min
Amount of V_2O_5 added (wt.%)	3, 5, 7 and 9

at a very slow rate in to the melt. The addition of magnesium promotes wettability of *in situ* generated particles and retains them inside the melt. The position of the stirrer is kept constant to disperse the V_2O_5 particles into the melt. The stirrer speed is kept at 800 rpm to form the vortex. After 15 min of stirring, the melt is poured in to a preheated cylindrical graphite mold of length 165 mm and inner diameter 45 mm. Degassing of the melt is carried out at this stage for vacuum casting. The cast *in situ* ingot is now allowed to cool in still air. Same parameters have been used for the entire *in situ* ingot with various wt.% of V_2O_5.

X-ray diffraction (XRD) using a D8 Advance from Bruker (Germany), using Cu Kα radiation, optical microscope, optical emission spectrometer was used for characterization. Before metallographic examination, Keller's reagent was used for polishing and etching purposes of the specimens. The optical microscope from Nikon (Japan) was used for the microstructure evaluation.

2.2 Dry sliding wear test

Tribological behaviors of various composites were investigated using a pin on disc tribometer from Ducom (Bangalore, India) as per standard test methods of ASTM G99-95. For the data acquisition system, a personal computer is connected with the tribometer. Linear variable differential transformer (LVDT) is used for determination of the wear in micrometers and sensors are mounted to sense the changes in the frictional forces. Weight of the pin was determined before the test was conducted and after the tested result. Weight loss of the pin was determined as a function of different load applied and sliding distances. Weighing was performed with an analytic balance Shimadzu AX 200 machine with a sensitivity of 0.01 mg.

Flat cylindrical pins of 10 mm diameter and 30 mm height were used for the test. The counter face disc has a maximum diameter of 165 mm and is made up of EN-31 steel hardened to 60 HRC. The thickness of the disc=08 mm and the limit of disc track diameter=145 mm. The chemical composition of EN31 steel is C 1, Si 0.35, Mn 0.5, S 0.05, P 0.05, Cr 1.3 and Fe balanced. Before conducting each experiment, ethyl alcohol was used to ensure that the surfaces were cleaned properly. The entire tests were conducted at ambient temperature with track diameter=40 mm (for each experiment). The loads applied were 10 N, 20 N and 30 N with sliding distance 1000 m. The sliding velocity for all of the experiments was 0.83 m/s. The samples were cleaned and polished with 400, 600, 800 and 1000 grit paper. Emery paper A350 was used for polishing the disc after each experiment. After completion of each test, the pin and disc specimen was cleaned ultrasonically with ethyl alcohol and stored in a vacuum oven furnace to avoid corrosion of the material. For the examination of the worn surfaces, a trinocular stereo zoom microscope was used. The mean average value was used after completing each experiment three times to maintain accuracy in the results.

3 Results and discussion

3.1 Thermodynamics characteristics of the *in situ* reaction

According to thermodynamic aspects, pure vanadium is formed by the reduction of V_2O_5 as shown in reaction (3). The vanadium formed reacts with excess aluminum and forms Al_3V as shown in reaction (4):

$$3V_2O_5+10\ Al\rightarrow 6[V]+5Al_2O_3 \tag{3}$$

$$\Delta G^0_{298}=\text{-3735 KJ/mol},\ \Delta H^0_{298}=\text{-3727 KJ/mol}$$

$$3Al+[V]=Al_3V \tag{4}$$

$$\Delta G^0_{298}=\text{-24.5 KJ/mol},\ \Delta H^0_{298}=\text{-20 KJ/mol}$$

The negative value of ΔG^0_{298} for both reactions shows that these reactions can thermodynamically occur and also that the reactions are exothermic. Based on high negative values, it seems that the *in situ* reaction occurred between Al and V_2O_5 during the melting process. Also, results obtained by XRD indicate that the reaction occurred without the formation of any intermediate products.

3.2 XRD analysis

The phases identified by the XRD technique are presented in Figure 3. Al_3V and Al_2O_3 were identified in Al-V_2O_5 composites. In case of 5% V_2O_5 addition, strong peak was observed in comparison to 3% and 7% addition due to complete reaction between Al and V_2O_5. Reduction of aluminum can be easily identified with decrement of the peaks with the increment of particle addition. Two phases appeared apart from aluminum in XRD, i.e. Al_3V and Al_2O_3; according to the Al-V phase equilibrium diagram $Al_{21}V_2$, $Al_{45}V_7$ and $Al_{23}V_7$ are metastable over a temperature of 736°C.

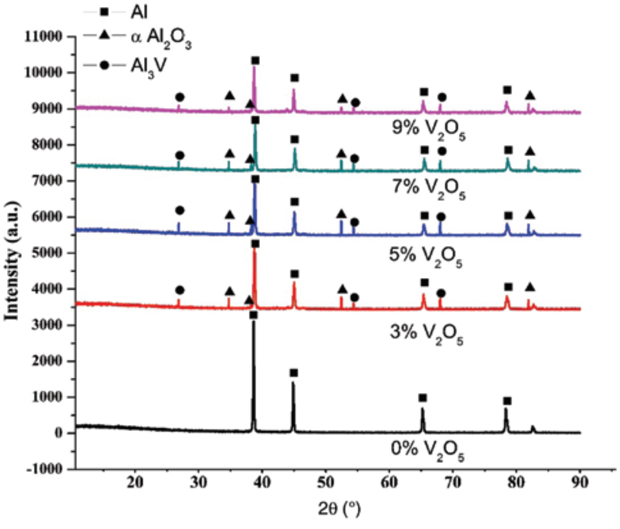

Figure 3: X-ray diffraction (XRD) pattern for cast *in situ* composite synthesized at 850°C.

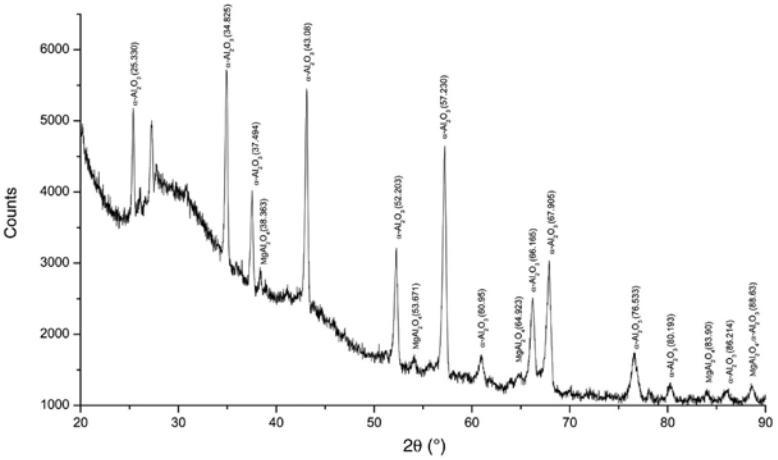

Figure 4: X-ray diffraction (XRD) pattern of filtered residue obtained from composite Al (5 g Mg)-5% V_2O_5, synthesized at 85°C.

Al_2O_3 formations due to the *in situ* reaction were verified by a simple chemical test. About (3.0 g) of V_2O_5 *in situ* composite samples were dissolved in hydrochloric acid (2 м) without attacking the unreacted V_2O_5 and Al_2O_3 particles [20]. For settling the fine particles, sodium sulfate (0.1 м) was used. The insoluble oxide particles were filtered out using Whatman ashless filter paper and the filtrate was heated up to 900°C for 1 h and allowed to cool down. After filtration, the residue is collected for the mass determination of alumina. The maximum recovery of the particles extracted was 78.21%. An XRD study was conducted using the Bruker (Germany) D8 Advance, with two theta range from 15 deg to 90 deg using Cu K_α radiation. The XRD of the cast 5% V_2O_5 composite is shown in Figure 4. XRD results clearly show the formation of alumina particles due to the *in situ* reaction between pure aluminum and V_2O_5 particles. The scanning electron microscopy (SEM) image of extracted particles is shown in Figure 5.

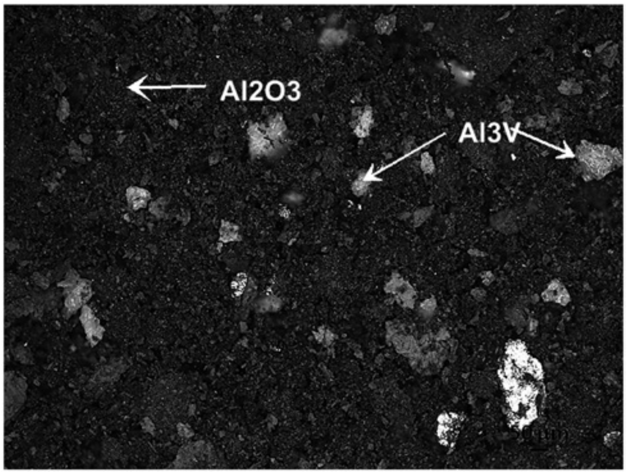

Figure 5: Scanning electron microscopy (SEM) image of extracted particles.

3.3 Microstructural evaluation

Micrograph of the Al with various percentage additions of V_2O_5 is shown in Figure 6. Figure 6A shows the microstructure of pure aluminum and Figure 6B and C indicate the grain refinement of aluminum alloy due to 3% and 5% addition of particles, respectively. Figure 6D shows the microstructure for 5% addition at higher magnification. The grain size of pure Al and the composite made by 5% addition was measured with the help of metallurgy plus software. The average grain size measured for Al was 1 µm. In the case of 5% composite, the average grain size was reduced from 1 µm. The reduction in the grain size was found to be due to the effect of vanadium. The grain size refinement indicates the homogeneous distribution of vanadium pentoxide particles in aluminum and formation of alumina due to complete *in situ* reaction. However, the particles are segregated at the grain boundaries due to pushing of particles at 3% composition, whereas at higher composition, i.e. at 5%, a complete reaction occurs which is shown in the microstructure. Al_2O_3 particulates are settled along the grain boundaries in a rod shape and a homogenous composite formed. In the case of 7%, a large number of oxide particles agglomerate at the grain boundaries and into the crystal structure.

3.4 Microhardness and tensile strength

Table 2 shows the mechanical properties of pure Al and various cast Al-V_2O_5 composites. Microhardness has been

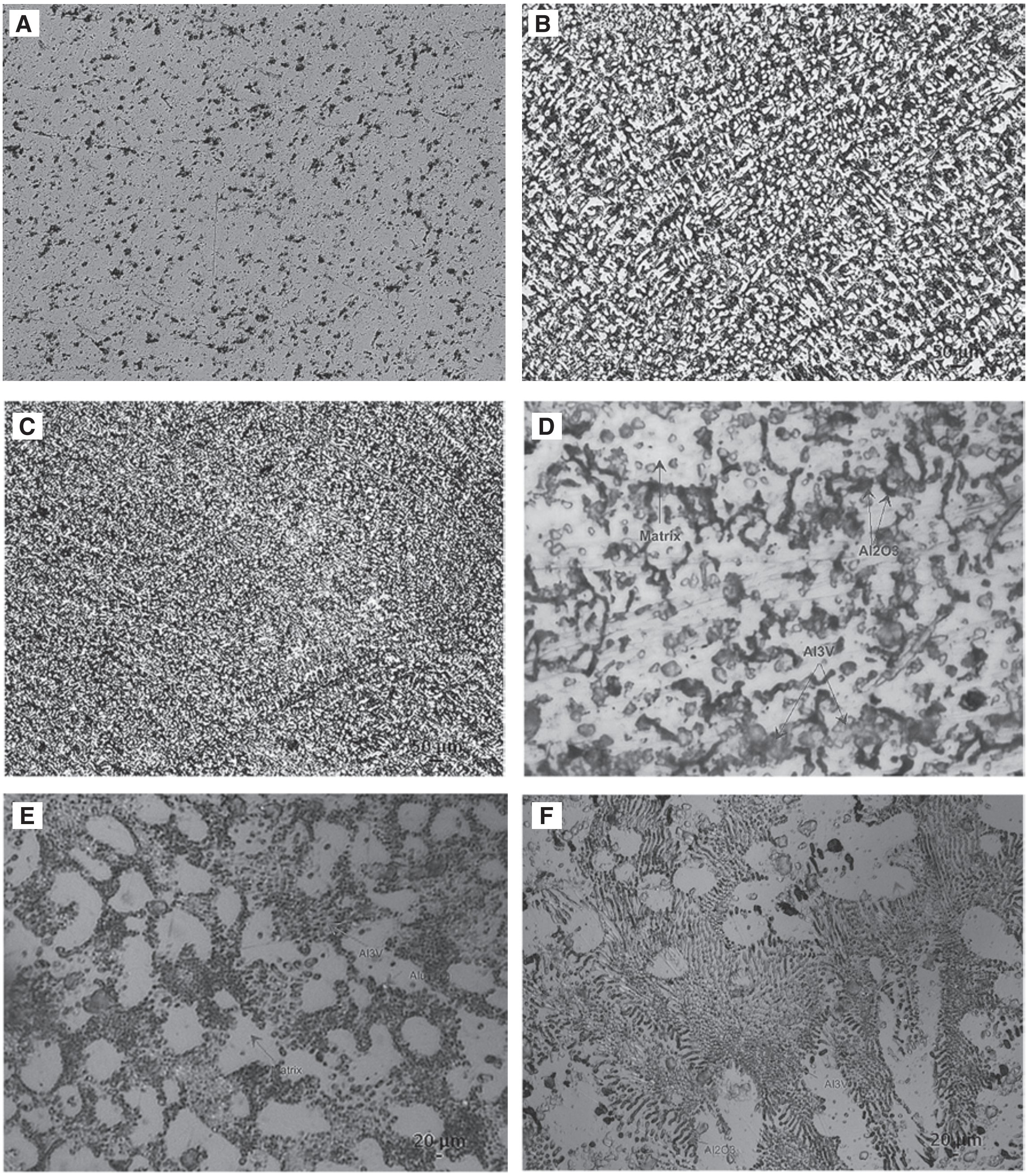

Figure 6: (A) micrograph of pure aluminum; (B) shows the micrograph Al-3% V_2O_5; (C) Al-5% V_2O_5 at low magnification; (D) Al-5% V_2O_5 at high magnification; (E) Al-7% V_2O_5 at low magnification; (F) Al-7% V_2O_5 at high magnification.

Table 2: Mechanical properties and porosity content for different reinforcements[a].

Sample[b]	Material	Microhardness (HV)	UTS (MPa)	Elongation (%)	Porosity content (vol.%)
A	Pure Al	25.4 (1.01)	39.1 (6.7)	3.25 (0.4)	2.730
AV3	Al-3% V_2O_5	58.2 (1.45)	57.3 (4.8)	5.73 (0.7)	2.420
AV5	Al-5% V_2O_5	62.3 (1.82)	64.8 (2.6)	7.12 (1.3)	2.100
AV7	Al-7% V_2O_5	58.7 (2.03)	60.7 ((3.5)	6.44(0.8)	3.040
AV9	Al-9% V_2O_5	47.8 (1.32)	49.4 (2.4)	4.21 (0.5)	3.810

[a] Standard deviations are shown within parentheses.

[b] A stands for aluminum, V stands for V_2O_5 and the last digit represents the wt.% of oxide added.

measured for the more than 20 region points at 200 gf load with 5 s dwell time.

A major increment in hardness is observed in the cases of AV3 and AV5. The increment in hardness is due to solid solution hardening, where V reduced from V_2O_5 and dissolved in molten metal. The solid solution hardening is the effect attained by adding the alloying elements into the matrix. The solid solution hardening is a result of an interaction between mobile dislocations and the solute atoms. The presence of solute atoms increases the initial yield stress and reduces the dynamic recovery rate of dislocations. According to Hall-petch, grain boundaries perform as a barrier for dislocation slip and refine grains provide more area to resist the movement of dislocation. The micrograph shows the grain refinement. The grain size refinement creates a high volumetric density of grain boundaries which impede the dislocation movement and propagation to adjacent grains, thereby consequently strengthening the material [21, 22]. Also, good distribution of V_2O_5 particles acts as an obstacle for the dislocation movement and forms the dislocation loops. According to the Orowan mechanism movement of dislocation is quite difficult in the case of a loop rather than a line [23, 24]. Dispersion of Al_2O_3 and intermetallic compound in the matrix also contributed to the hardness increment.

Figure 7 shows that sample AV5 indicates the maximum microhardness compared with other samples. Further addition of V_2O_5 results in decrease of hardness. The reason for hardness decrement is dendritic growth and release of latent heat. Agglomerations of a large number of particles in the crystal and at grain boundaries were also responsible for hardness decrement. AV7 has low density as compared to the pure Al and indicates the presence of porosity which is also a reason for low hardness.

The tensile strength of AV5 is higher than that of the other composites as well as pure aluminum. Strengthening is possible due to the presence of Al_2O_3 and V particles in the matrix.

Figure 8 shows the tensile fracture surfaces of cast composites with 5% and 7% addition of V_2O_5. The fracture surface shows some dimple ductile fracture in some areas as shown in Figure 8A, but also certain a area represents the shear fracture which results in the elongation of about 7%. Figure 8B shows the presence of dimples and the porosity (3.04). The presence of porosity is found to be responsible for the decrease in hardness as mentioned in Table 2.

Ductility plays an important role and affects the mechanical properties. Table 2 shows the ductility value with various percentages of V_2O_5 addition. Ductility increases until the 5% reinforcement, therefore tensile strength also increases. Higher reinforcement of more than 5% increases the porosity content as shown in the

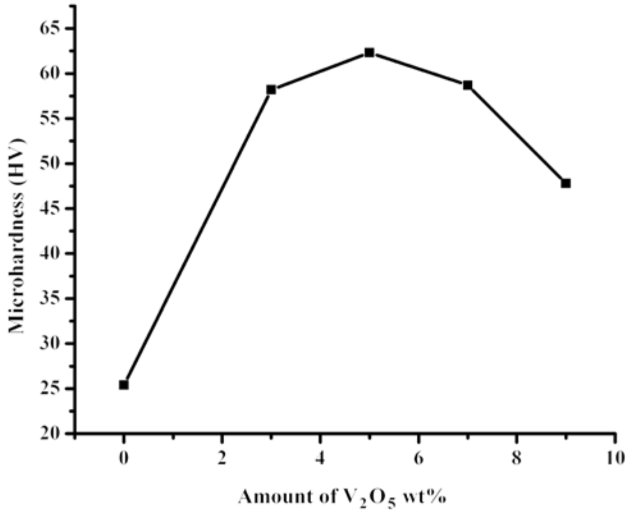

Figure 7: Variation of microhardness with amount of V_2O_5 added.

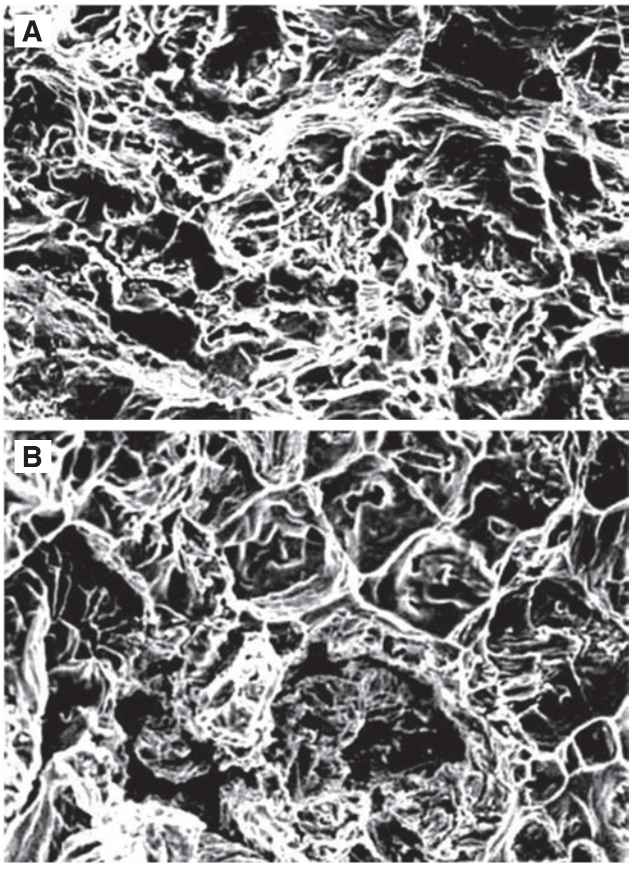

Figure 8: (A) scanning electron microscopy (SEM) showing the tensile fracture surface of composite with 5% addition; (B) SEM image of tensile fracture Al-7% V_2O_5.

SEM image. In the case of 7%, less tensile strength is reported due to porosity.

3.5 Coefficient of friction analysis

Figure 9 shows the variation of friction coefficient with respect to sliding distance at different loads, i.e. 10 N, 20 N and 30 N which was applied at a sliding velocity of 0.83 m/s. Generally, friction coefficient increases with increase of load. It is observed from the figure that the maximum coefficient of friction was shown by the pure aluminum. There was a significant decrease in the coefficient of friction with the addition of oxide particles of V_2O_5. A decrease of about 4.18%, 5.17% and 12.78% was observed in the coefficient of friction for composites containing 3 wt.%, 5 wt.% and 7 wt.%, respectively, in comparison to pure aluminum. The significant decrease in the coefficient of friction can be attributed to the formation of a lubricating layer between the metals in contact. This tribo layer formation was due to smaller particle size of V_2O_5. Lower value of coefficient of friction was due to proper dispersion of particles into the aluminum matrix [25].

A comparative plot was measured in the steady state region for coefficient of friction with respect to load, as shown in Figure 10. As stated earlier, coefficient of friction increases with increase of load and composites with 7 wt.% V_2O_5 had minimum coefficient of friction. The coefficient of friction was decreased around 10% in the case of 3% and 5 wt.% addition, whereas in case of 7 wt.% addition 20% decrement was observed at 10 N load. At 20 N loads, a significant decrease was observed in the coefficient of friction due to the addition of 3 wt.%, 5 wt.% and 7 wt.%, which was about 5.5%, 16.6% and 19.4%, respectively.

3.6 Volume loss

Figure 11 shows the variation of pin volume loss as the function of load for 1000 m sliding distance (steady state regime). A decrease in volume loss was observed in the cast *in situ* composite as compared to pure aluminum. The comparison of volume loss at 10 N, 20 N and 30 N with sliding distance 1000 m is shown in Figure 9. At the highest load (30 N), a reduction in volume loss was observed in as cast *in situ* composites as compared to pure aluminum. The volume loss observed was about 20%, 33.3% and 46% for Al 3%, Al 5% and Al 7% cast

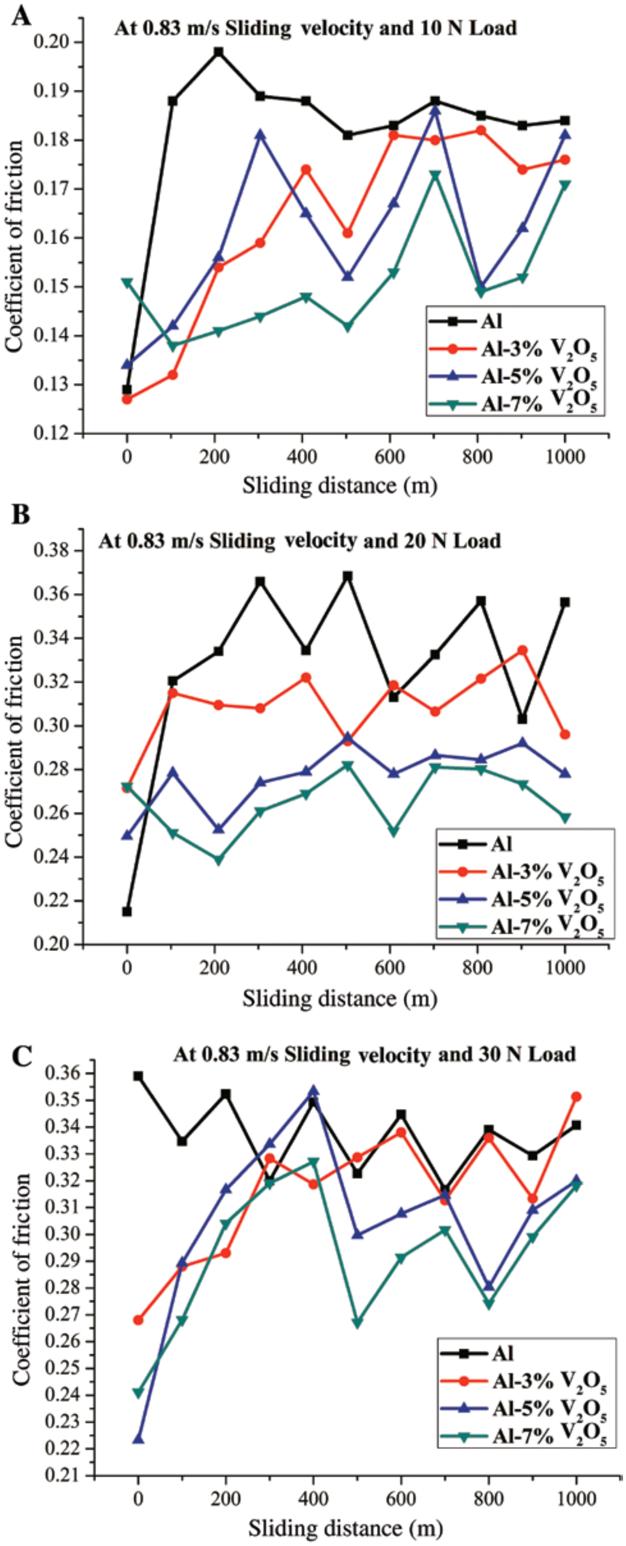

Figure 9: (A) Coefficient of friction as a function of sliding distance at 10 N load; (B) coefficient of friction as a function of sliding distance at 20 N load; (C) coefficient of friction as a function of sliding distance at 30 N load.

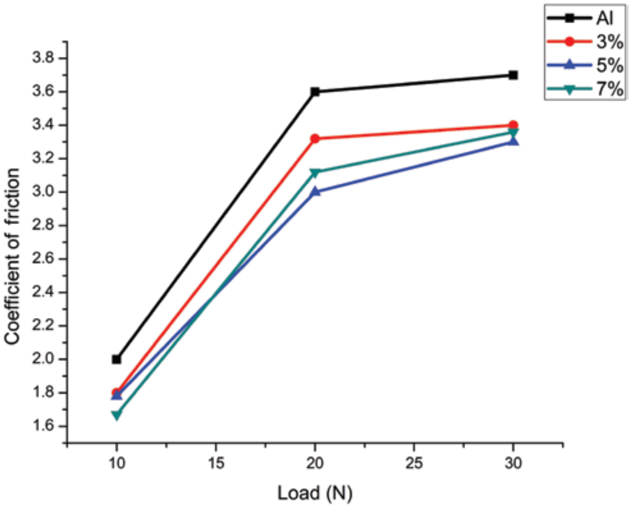

Figure 10: Coefficient of friction as a function of various loads.

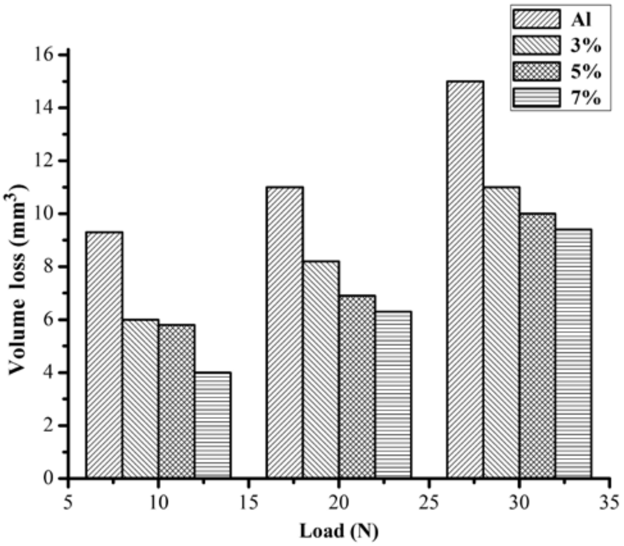

Figure 11: Relationship between volume loss at different loads for cast *in situ* composites.

samples, respectively. The porosity content in cast composites was a little higher than that in pure aluminum. Therefore, the reduction in volume loss in *in situ* composites over the pure aluminum could be attributed to the effect of reinforcing particles in the aluminum matrix.

3.7 Wear

Figure 12 shows the wear behavior of pure aluminum and cast composites with addition of V_2O_5. It is well known that the dry sliding wear of *in situ* composites decreases significantly by increasing the amount of reinforcing particles [26]. However, some researchers reported that the cast composites contain porosity due to increase in particle content, which tends to decline their behavior against wear. The porosity of the cast composites was found to be due to the poor wetting of the reinforced particles in aluminum melt [27].

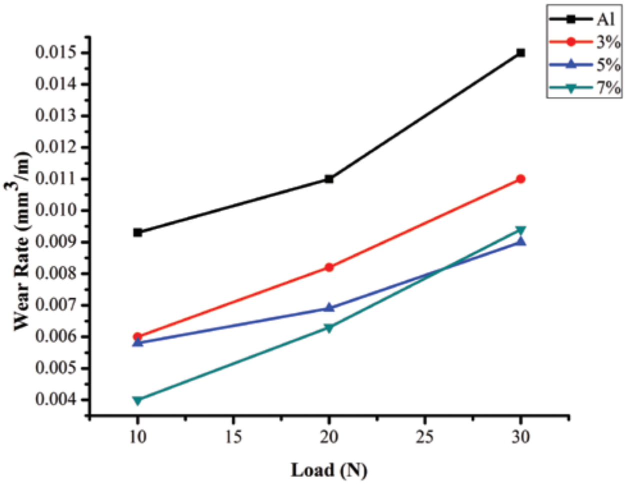

Figure 12: Wear rate of pure Al and cast *in situ* composite as a function of load.

From Figure 12, it was observed that with higher percentage of V_2O_5 particles wear decreases. With the addition of oxide particles, hardness increases and decreases the real area of contact. During the dry sliding test, the oxide debris was generated. This hard debris locked between the surfaces and promotes wear. However, at the same time, particles get compacted between the surfaces and form a protective transfer layer. This layer is responsible for the decrease in wear rate [28]. However, for 7% addition specific wear rate tends to increase. This increase was due to the presence of porosity in cast *in situ* composites. The porosity also affects the hardness and tensile properties as reported in Table 2. The real area of contact increases with higher porosity and eventually increases the wear rate. So, the effect of porosity is not only softening the material, but it also promotes the delamination and subsurface cracks.

The worn surface of material mainly depends on speed and applied load. At low load due to the micromachining effect, a proportion of counter face material is removed and subject to oxidation. This oxidized material (Fe_2O_3) acts as a tribo layer between the two surfaces. Wear studies reported that the wear rate temporarily reduces due to the tribo layer formation. This tribo layer acts as a solid lubricant between the mating surfaces.

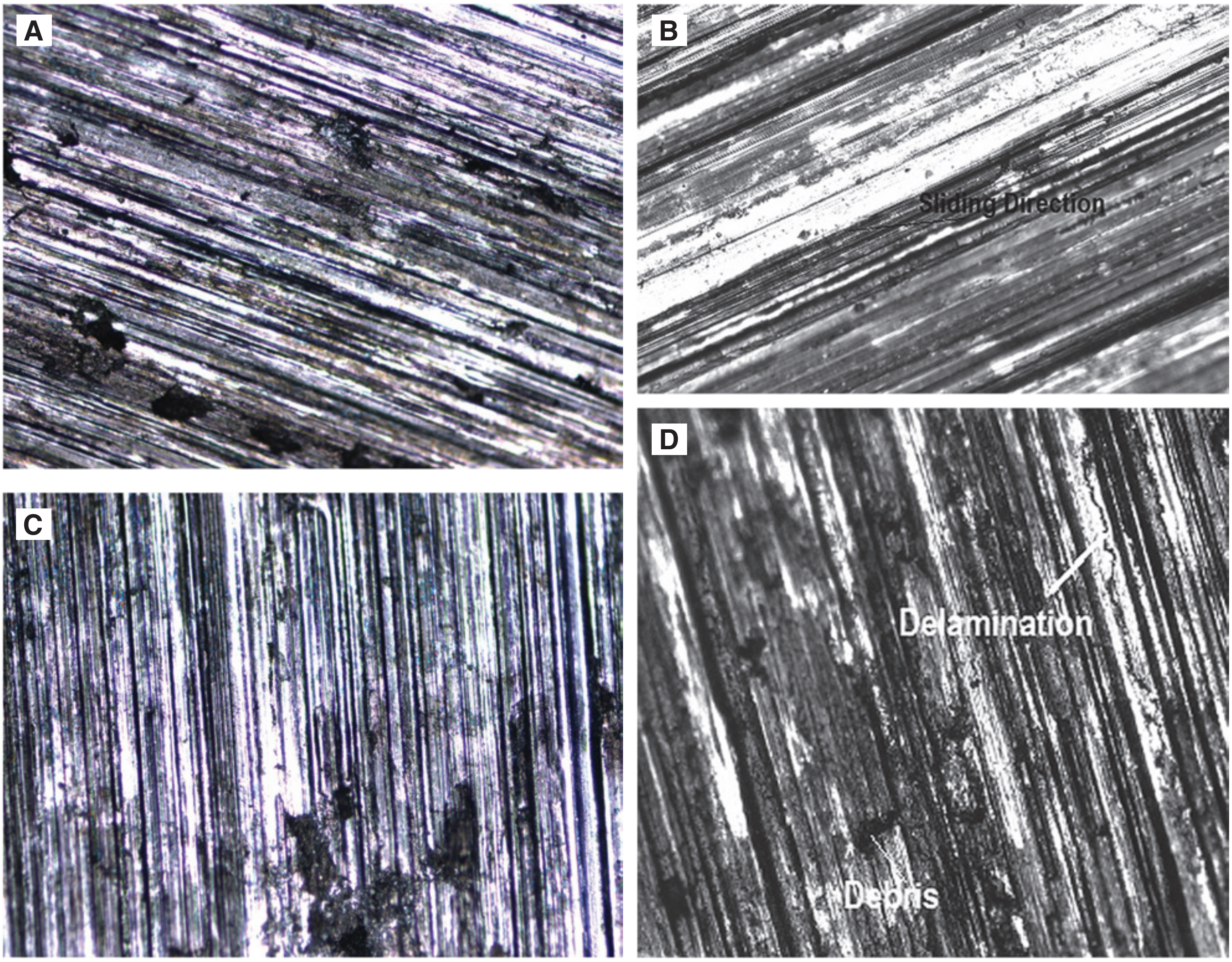

Figure 13: Micrographs of (A) worn surface for 5% at 20 N load; (B) worn surface for 5% at 30 N load; (C) worn surface for 7% at 20 N load; and (D) worn surface for 7% at 30 N load.

At high load, delamination was found as shown in the figure due to the removal of the tribo layer. At high load, high temperature is generated at near surfaces and reduces the shear strength in subsurface layers which promotes the excessive material transfer. The best result of wear rate has been found in the case of 5% addition as compared to the 7% addition. Micrographs of worn surfaces at different loads with 5% and 7% addition are shown in Figure 13. At low load, small grooves were observed on worn surfaces in the sliding direction due to an abrasion mechanism. However, in the case of higher load, delamination occurs as the real area of contact increases and eventually the fracture stresses of reinforced particles increase. The presence of alumina and intermetallic compound in the cast *in situ* composite provides hardness, but if the porosity increases, interaction between the matrix and reinforcement decreases.

4 Conclusion

An *in situ* aluminum metal matrix composite was developed using pure aluminum ingot and V_2O_5 particles. The microstructure and effects on microhardness were measured and analyzed. The dry sliding wear behavior of aluminum and cast *in situ* composites containing different wt.% of oxide particles was investigated. The effect of oxide particles addition and porosity on wear was evaluated. The conclusions were summarized as follows:

1. An *in situ* composite was formed due to the reduction of V_2O_5 particles by molten aluminum as revealed by the XRD study. Aluminum melt liberates the vanadium ions first, which further reacts with molten aluminum and forms an Al_3V intermetallic compound. The extent of reduction increases with increasing the percentage of oxide particles up to 5%.

2. Increment in microhardness is observed to be approximately 2.5 times more than that of pure aluminum in the case of composites formed by the 5% addition of V_2O_5. The reaction found in this case is nearly complete and formation of Al_2O_3 and Al_3V was found to responsible for improvements in hardness and microstructure.
3. Vanadium acts as a grain refiner and in the microstructure, grain refinement is observed. From the experiments it was observed that the addition of a small amount of V_2O_5 particles gives appreciable results.
4. The coefficient of friction value was found to be low in the case of *in situ* composites as compared to pure aluminum.
5. Wear rate of *in situ* cast composites seems to be lower in comparison to aluminum. In the case of 7% additions of oxide particles, more wear was observed.

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