MICROSTRUCTURE CHARACTERIZATION OF AL/ CALCINED DOLOMITE METAL MATRIX COMPOSITES

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Abstract
Al/calcined dolomite metal-matrix composites (MMC) were fabricated and tested by varies the calcined dolomite content (10-30 wt%) and using unidirection compaction and sintering as consolidation method. Upon incorporation of calcined dolomite in pure aluminium base metal, the properties of the resultant composite have been enhanced. The composites are lighter than the base metal and it also have high thermal stability due to its high melting temperature compared to base metal. Light materials with high degree of hardness and high thermal stability are prominent materials for wide range of applications. Microstructural studies of the composites have been performed using optical microscope and Scanning Electron Microscopy (SEM) and will be described in this paper.

Keywords: Al/calcined dolomite, powder metallurgy, metal-matrix composites (MMCs), microstructure.

Introduction

Particle reinforced aluminum matrix composites (AlMCp) possess high-specific elastic modulus, high-specific strength, good wear resistance and excellent properties at elevated temperature over conventional aluminum. They are considered as potential lightweight and high performance materials to be used in aerospacecraft, aircraft and engine parts in automobiles [1]. The renewed interest in metal–matrix composites has been aided by the development of reinforcement material, which provides either improved properties or reduced cost when compared to the existing monolithic material. The marketed supports are generally manufactured Al composite reinforced with ceramic particulate such as Al_2O_3 and SiC and etc which have relatively higher in cost [2]. In order to decrease the cost and to utilized our natural resources, in this work, calcined dolomite powders was synthesized from the mineral dolomite (CaMg(CO3)2) and have beed used as a reinforcement in aluminum composites. Sintered dolomite (doloma) had gain importance in the steel-making industry due to its high thermomechanical and erosion wear properties [3].

Experimental

Materials and processing

Calcined dolomite which consists of CaO and MgO phase had been obtained from dolomite powder which had beed calcined at 1100 ◦C for 4 h. Figure 1 shows XRD spectrum of calcined dolomite. The purity of the added calcined dolomite obtained by fusion bead XRF is about 99.65 wt%. It contains, mainly, 0.35 wt% Al_2O_3, as purities. Commercial aluminum powder (99.0% purity) with 10 and 20 and 30 wt% of calcined dolomite powders have been mixed in a planetory ball mill with zirconia balls at 300 rpm. Then the mixed powders are cold pressed at 250 MPa to form cylindrical green compacts with a diameter of 12 mm. The compacts have been heated at temperature of 610°C in nitrogen atmosphere.

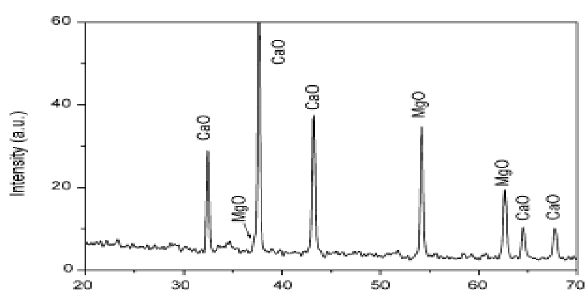


Figure 1: XRD spectrum of calcined dolomite powder after calcination at 1100 °C for 4 h

Table 4.0: Raw materials and particles size.

Raw materials	Average particles Size
1. Aluminum powder	26.181 µm
2. Calcined dolomite (4h calcined at 1100°C)	21.474 µm

Table 4.1: Raw materials after mixing and particles size.

Sample	Composition	Average particles Size after milling
ALCD10	Aluminum + 10wt% of calcined dolomite	25.636 μm
ALCD20	Aluminum + 20wt% of calcined dolomite	25.776 μm
ALCD30	Aluminum + 30wt% of calcined dolomite	26.066μm

Material characterizations

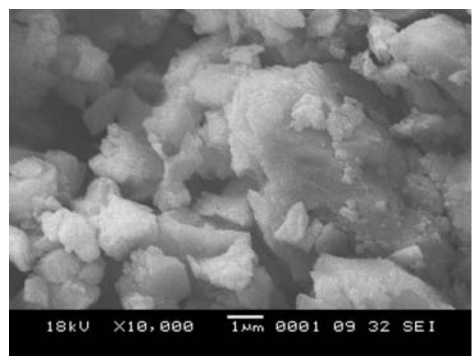
The polished samples that were etched with Keller's reagent were examined under digital microscope and Scanning Electron Microscope model JEOL-JSM6064LA fitted with EDX. All the samples were sputtered with copper prior to SEM examination. Secondary electron and backscattered electron was used on samples to determine the distribution of the reinforcement and porosity.

Results and Discussion

Characterization of the initial materials

The starting powders were examined by Scanning Electron Microscopy (SEM) and the resulting micrographs are illustrated in Figure 1 and Figure 2. The microstructure aluminum powder was in spherical shape at 1,000 X magnification wheras the calcine dolomite was irregular in shape and the micrograph was taken at 10,000X magnification. Particle size of calcined dolomite is lower than Al, however it has wide range of size. Mechanical milling of mixed powder at 300 rpm for 2 hours didn't give significant effect in reducing the average particle size of the powders composites.However the multimodal distribution of reinforcement partical was changed to monomodal distribution. In addition, the powders were well mixed and this can be seen in micrograph of green compact sample as shown in figure 5.

(a)



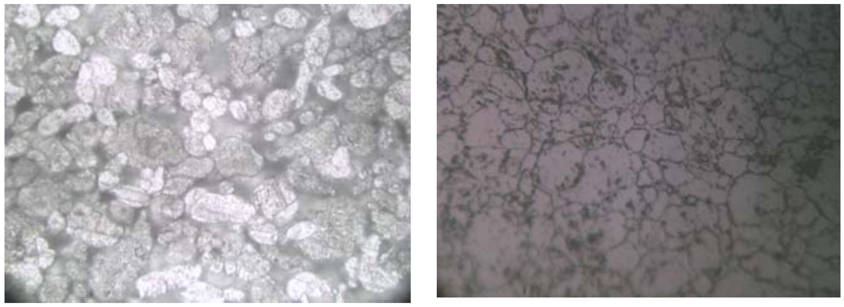
(b)

Figure 2: SEM micrograph of initial raw materials; (a) pure aluminium. (b) calcined dolomite

Microstructure of green compact and sintered samples

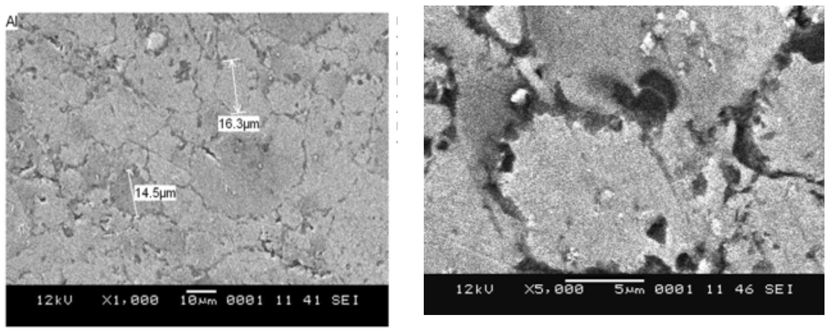
Figure 3 are represant green compact sample and sintered sample of pure aluminum obtain by digital microscope while figure 4 shows sintered Al at high magnification using SEM. This aluminum continuous phase consolidation is used as a reference data to measure the change occuring as a result of the addition of the calcined dolomite. Etched sample of aluminum reveal equiax grains. At high magnification, the presence of void or porosity can be clearly seen especially at tripple grain point. This demonstrates that high degree of compaction was not achieved in the aluminum continous phase matrix during consolidation. The pore structure is open and interconnected and the pore morphology is not very smooth due to the existence of relatively sharp curvature gradients near the interparticle neck region.

Figure 5 shows the comparison microstructure between green compact samples and for sintered sample composites for different reinforcement contents by using optical light microscope at 600x magnification. Grain boundaries can be seen for samples that are sintered using nitrogen compared to the samples that are not being sintered. At first stage, the particle contacts are transformed to sintered bridges, called 'necks'. The contacts also exist for the samples that were not sintered in the form of microplanes instead of point contacts where their extension dependent upon compacting pressure. Grain boundaries are formed between two adjacent particles in the plane of contact. At intermediate stage, after strong neck growth, the single particles begin to loose their identity. A coherent network of pores is formed and grain growth occurs, which results in a new microstructure and the grain boundaries usually run from pore to pore [4].



(a) (b)

Figure 3: Micrograph of green compact Al and sintered Al using digital microscope at 600x magnification; (a) Green compact Al and (b) Sintered Al



(a) (b)

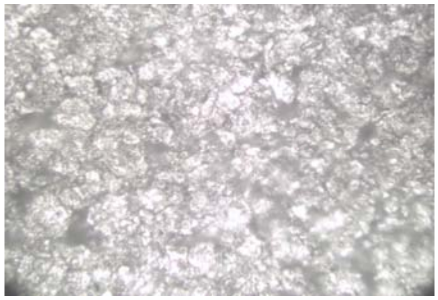
Figure 4: Pure Aluminum, sintered at 610°C in Nitrogen atmosphere a) 1000X b) 5000X

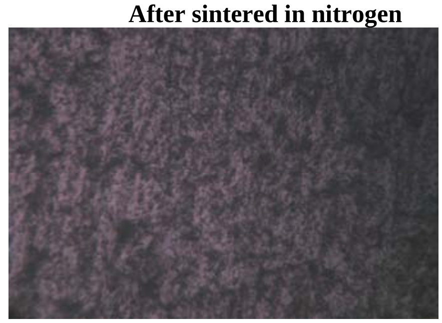
Metallographic examinations of the composite materials using optical microscopy also revealed the uniform distribution of calcined dolomite reinforcement particle in the Al matrix. The higher the weight percent of the reinforcement indicates the higher the black region. Figure 6 shows that at higher magnification, the agglomeration of calcined dolomite particles occurs at the grain boundaries of aluminum grains demonstrating a pore free surface. Micrograph also indicated that the interparticles regions were palstically deformed to accommodate the reinforcement. EDX analysis shown that part of the matrix was melted thus allowing it to diffuse into calcine dolomite reinforcement, where this might promote binding within Al and the reinforcement. The agglomerates seem to fill the spaces in between the Aluminum grains which contribute to miminal porosity characteristic. The continuous phase in this system deforms mainly by the localized melting.

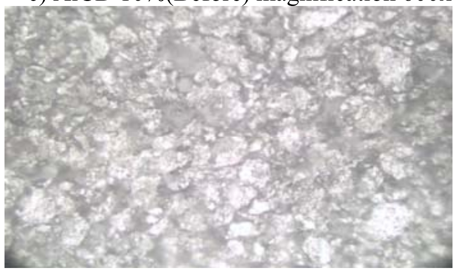
Micrograph of the samples Al/CD10 at figure 7 shows that calcined dolomite particle is bonded well to the Al matrix, since no structural defects, large voids or cracks are observed in the Al/calcined dolomite interface. Furthermore little particle pullout was revealed by image analysis techniques.There is also no apparent segregation of the calcined dolomite particles. Figure 8 shows that there are apparently three phases present in the microstructure, characterized by white areas, black areas and grey-black areas respectively. Once the matrix has wet the reinforcement it is in intimate contact with the reinforcement and bonding occurs. Strong inter-phase region determined the performance of the composite where it transfers the properties (e.g stress) from the matrix to the reinforcement.

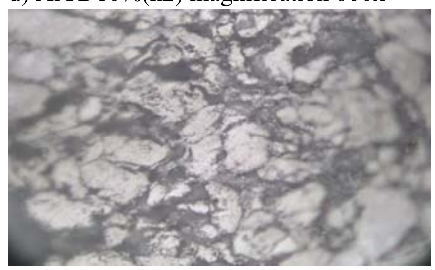
Chemical composition analysis was performed using energy dispersive X-ray spectroscopy. Figure 9 shows micrograph Al reinforced with 20wt% calcined dolomite. The EDX spectrum as show in figure 10 indicates the composition in the spot area. At spot point 010 at bright area shows only spectrum of Al peak present. Spot 011 at black area indicates elements like Ca, Mg, O, Al and C are present. The existance of Ca, Mg, O indicates that calcined dolomite are located in both intergranular and intargranular locations. The existance of Al in reinforcement area shows the part of matrix was melted, thus allowing Al to diffuse into calcined dolomite reinforcement where this might produce the binding together of calcined dolomite particles. In other word that calcined dolomite particles has react with aluminum matrix forming an interfacial phase. Elements like C, O also present due to some of carbonate which are not decomposed during calcination process of raw dolomite.

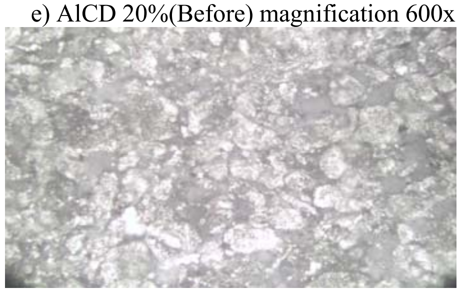
Before sintered **After sintered in nitrogen**


c) AlCD 10%(Before) magnification 600x


d) AlCD10%(n2) magnification 600x


e) AlCD 20%(Before) magnification 600x


f) AlCD20%(n2) magnification 600x


g) AlCD 30%(Before) magnification 600x

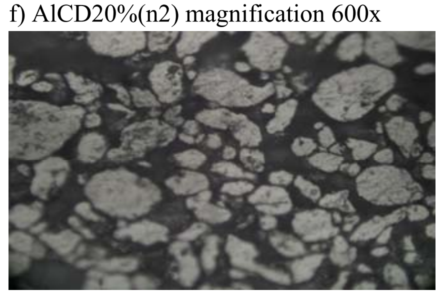
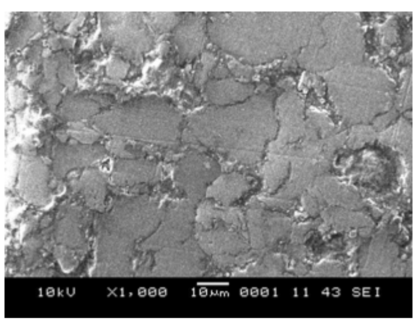

h) AlCD30%(n2) magnification 600x

Figure 5: Green compact and sintered composites Al with different wt% of calcined dolomite



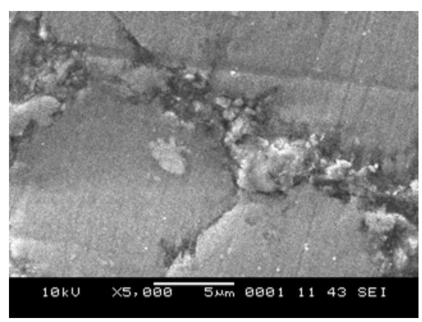
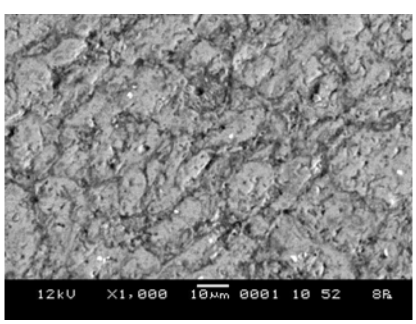


Figure 6: Al/20CD, sintered at 610°C in Nitrogen atmosphere (a) 1000X,. (b) 5000X



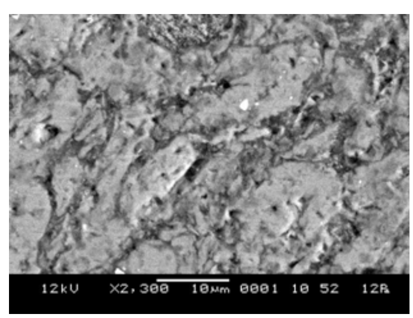


Figure 7: Al/10CD, sintered at 610°C in Nitrogen atmosphere a) 1000X b) 2300X shows elongated grain

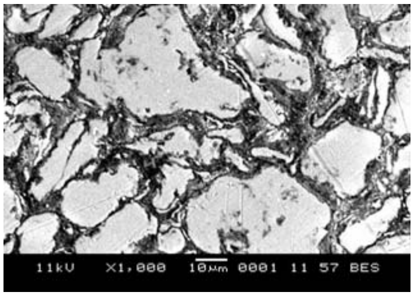


Figure 8: SEM/BSE of AlCD30 showing 3 phases present.

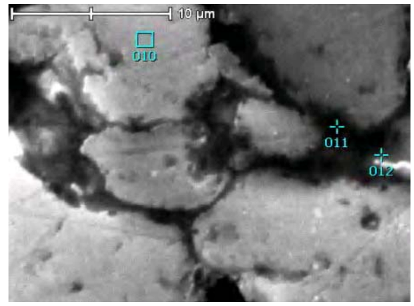


Figure 9: SEM/ SE of AlCD20

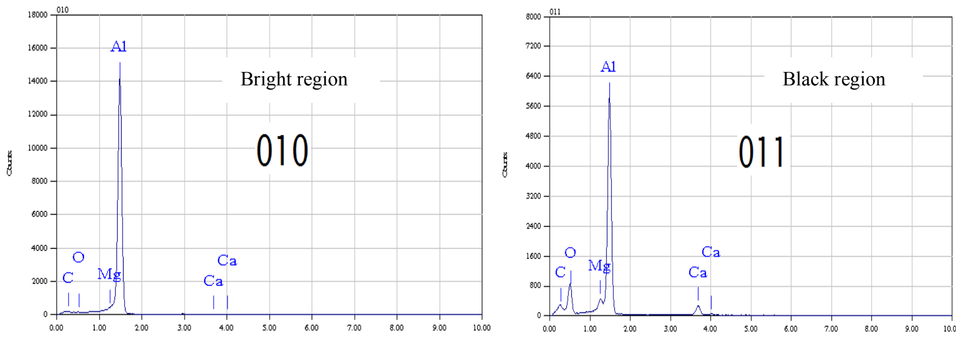


Figure 9: Result of EDS spectrum and composition for AlCD20 sample sintered in nitrogen.

Conclusion

After mixing, compaction and consolidation, micrograph of the sintered sample shown that calcined dolomite particles randomly distributed within the matrix or at the matrix grain boundaries. The addition calcined dolomite tends to reduce pore that are exist between Al grains. Futhermore it will distort the lattice and generate stress and will produce a barrier to dislocation motion. Prolonged sintering and high sintering temperature caused the matrix to melt. Futhermore the evolution of carbonate gas at certain area tends to deteriorate the microstructure of composite where some of the reinforcement was pulled out. SEM/BSE micrograph also shown that interphase between calcined dolomite and matrix Al was formed which results in good bonding between matrix and the reinforcement.

Reference

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