## 学位論文要旨

## Fabrication and Properties Evaluation of VGCFs/Mg-Al-Ca Composites (VGCFs/Mg-Al-Ca 複合材料の作製及び特性評価)

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Mg-Al-Ca alloys have been developed for elevated temperature applications. The addition of calcium significantly improves their high-temperature strength, creep resistance, and oxidation resistances. To enhance the strength and rigidity of the matrix, carbon nanofibers can be used to reinforce Mg-Al-Ca alloy, which are supposed to give them satisfactory mechanical properties at both room and elevated temperatures.

The cost-effective processing of composite materials is an essential element for expanding their applications. Among the variety of manufacturing processes available for discontinuous metal matrix composites, stir casting is easily adaptable and economically viable. Its advantages lie in its simplicity, flexibility, and applicability to large quantity production. However, there are some problems associated with stir casting of metal matrix composites such as: poor wettability and heterogeneous distribution of the reinforcement material.

The poor wettability of the reinforcement in the melt prevents the reinforcement from infiltrating the molten matrix, with the result that it simply floats on the melt surface. This is due to the surface tension, very large specific surface area, and high interfacial energy of the reinforcements, along with the presence of oxide films on the melt surface. Compo-casting is a liquid state process in which the infiltration of reinforcement into a semi-solid metal (SSM) can be facilitated by means of agitation.

The uniform distribution of the reinforcement within the matrix and its bond strength with the matrix are essential structural requirements for a stronger metal matrix composite. The wettability and distribution of the reinforcement are difficult because of the small size of VGCFs. This is due to the large surface area and surface energy of the particles, which cause an increasing tendency for agglomeration. Thus, an intermediate layer of nickel is coated on the carbon fibers to facilitate the wetting.

The final purpose of the present research is to develop the VGCFs reinforced Mg-Al-Ca alloy composites with satisfactory mechanical properties by the cost-effective fabrication process, to face the elevated temperature application. Prior to fabricate the VGCF-reinforced magnesium-calcium alloy composites, the effect of nickel coating on the wettability of magnesium with carbon fiber was investigated. Ni-coated VGCFs reinforced Mg-Al-Ca alloy composites were fabricated by compo-casting process. The effects of Ni-coated VGCFs on the microstructure and mechanical properties of magnesium alloy were investigated, and the strengthening mechanism of Ni-coated VGCFs reinforced Mg-Al-Ca alloy composites was discussed. The conclusions of this thesis are summarized as follows:

1. The study on the effect of nickel formation on graphite sheet surface for improving wettability with magnesium alloy (Chapter 2).

Prior to the fabrication of VGCF/Mg-Al-Ca composites, the wettability of magnesium alloys with graphite sheet, which has the basal plane of (002) same with that of VGCFs, was studied, and the effect of nickel coating on wetting behavior of magnesium alloys and graphite sheet was investigated. Mg-Al alloys did not wet graphite sheet with the contact angle of about 120°. The droplet of magnesium alloy on Pure Ni spread rapidly. By means of the wetting of Mg-Al-Ca alloys on pure nickel, the mechanism for the wetting behavior between liquid magnesium alloys and solid nickel was proposed. Nickel dissolved into magnesium alloy during contacting. Spreading of the droplet stopped when nickel saturation occurred and Mg<sub>2</sub>Ni intermetallic compound began to form. Improvement of wettability of magnesium alloy on Gr-Ni was achieved through the dissolution of nickel into the liquid magnesium alloy at the interface between droplet and substrate. Calcium addition had no effect on the wetting of magnesium alloy on Gr, while it

showed negative effect on the Pure Ni, and slightly hindered the spread of magnesium alloy droplet on Gr-Ni.

2. The study on the fabrication of VGCF-reinforced magnesium matrix composites by low pressure infiltration (Chapter 3).

The effect of nickel coating on infiltration behavior of magnesium alloy into VGCF preform was investigated. Fabrication of VGCF-reinforced magnesium alloy composites was carried out by low pressure infiltration. Infiltration pressure for magnesium alloy into porous VGCF preform was determined to be 1 MPa, considering with both the estimated threshold pressure and the compression strength of the preform. Magnesium alloy was successfully infiltrated into nickel coated porous preform, while the magnesium alloy was partially infiltrated into the preform without nickel coating.

3. The study on the fabrication of VGCF-reinforced Mg-Al-Ca alloy composites by compocasting process (Chapter 4).

Microstructure of Mg-5Al alloy consisted of  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> intermetallic phase in the  $\alpha$ -Mg matrix. With the additions of calcium, the Mg-Al-Ca alloy exhibited a dendritic microstructure with calcium-containing phases along the grain boundaries which suppressed the formation of the Mg<sub>17</sub>Al<sub>12</sub> phases. With increasing Ca/Al ratio, the volume fraction of the second phase in the ascast Mg-Al-Ca alloys increases, and the main second phase is (Mg, Al)<sub>2</sub>Ca with a coarse lamellar structure.

Nickel-coated VGCFs were prepared using an electroless plating process, with the nickel homogeneously deposited on the VGCFs. Ni-coated VGCF-reinforced Mg-Al-Ca composites were fabricated using the compo-casting method. The preliminary mix of the Ni-coated VGCFs into the semi-solid metal was facilitated by compo-casting process. With the nickel coating diffused into the metal, the dispersion of VGCFs was promoted. Al<sub>3</sub>Ni compounds formed both inside the grains and on the grain boundaries. The present of Al<sub>3</sub>Ni compounds and VGCFs disturbed the reticular distribution of (Mg, Al)<sub>2</sub>Ca along grain boundaries. The addition of Ni-coated VGCFs could refine the grain of the Mg-Al-Ca alloy.

4. The study on the mechanical properties of VGCF-reinforced Mg-Al-Ca alloy composites (Chapter 5).

With Ca content increasing, the Mg-Al-Ca alloy exhibits a higher yield strength and strainhardening than the Mg-5Al alloy. However, the reticular distribution along grain boundaries of the (Mg, Al)<sub>2</sub>Ca phase results in the impairment of elongation at room temperature, which also affect the ultimate tensile strength. The obvious elevated temperature strengthening effect was obtained by calcium addition, since (Mg, Al)<sub>2</sub>Ca has much high melting point and thermal stability. The reduction of tensile strength of Mg-Al-Ca alloys with temperature increasing declines. However, with Ca content increasing, reticular (Mg, Al)<sub>2</sub>Ca formed along grain boundaries results in the decrease of elongation or a little change of tensile strength at elevated temperature.

With a 0.5 wt% addition of Ni-coated VGCFs, an increase in the UTS of the composite was achieved, along with an increase in the total elongation, which could mainly be attributed to the strain hardening during a larger strain. However, for the 1.0% VGCF-reinforced AX53 alloy composites, the elongation dropped, which led to a low strength similar to that of the AX53 matrix alloy. The addition of VGCFs in AX53 alloys shows some strengthening effect at elevated temperature. The strength and ductility of the composite are improved simultaneously.

The contributions of load transfer, Orowan strengthening, thermal mismatch and Hall-Petch effect were simply added up to predict the potential yield strengths by adding different weight fractions of VGCFs to the composites. In this model, load transfer and thermal mismatch strengthening are the main mechanism to the improvement of the yield strength. The experimental result of yield strength was compared to the theoretical value. It showed that the dispersion still needs to be improved to produce a stronger material.