Fabrication and Characterization of Squeeze Cast AC4A Al/TiNi Shape Memory Composites

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SUMMARY: Reinforcement of shape memory alloys within light weight metal matrix reveals compressive residual stresses in the matrices resulting in improved tensile properties of the composites. In the present study, Al/TiNi shape memory composites were fabricated by the squeeze casting technique. Optimal fabrication conditions for squeeze casting were obtained experimentally. Fairly good microstructure and matrix/reinforcement bonding observed in the squeeze cast Al/TiNi composites. Tensile tests of the composites were carried out above A_f (austenite finishing temperature) which was found from DSC analysis. The amount of prestrain to induce the compressive residual stress in Al matrix and the recovery force of the TiNi were determined from tensile curves of TiNi wire. Improved strength of the composite due to TiNi reinforcement as well as compressive residual stresses in Al matrix was identified. It was found that the total interfacial area between matrix and reinforcement which depends on the wire diameter also affected the strength enhancement.

KEYWORD : interfacial bonding strength, compressive residual stress, stress-induced martensite, shape memory recovery, Al/TiNi composite.

INTRODUCTION

Al, Mg, Ti alloy metal matrix composites reinforced with ceramic fibers have good properties such as low specific gravity, high specific strength and stiffness. They are being expected as materials of automotive parts because of their good wear resistance, thermal property and low thermal expansion coefficient(CTE) [1],[2]. These potential materials, however, have demerits such as the interfacial weakness due to their poor chemical attraction and wettability between the metal matrices and ceramic reinforcements, high manufacturing cost and complexity of production processes. Moreover, tensile strength and mechanical properties of composites are deteriorated by the tensile residual stress in the matrix which is caused by the CTE mismatch between metal matrix and ceramic reinforcement during the cooling process. Therefore, in the present study, shape memory composite(SMC : AC4A Al/TiNi) was chosen to solve problems of metal/ceramic composites. Recovery force of prestrained shape memory alloy upon heating above A_f gives compressive residual stress in the matrix and it can improve mechanical properties of composite such as strength and crack close [3].

The effect of shape memory alloy reinforcement on the mechanical properties of SMC was evaluated by the process described in Fig. 1 [4],[5]. This figure shows the enhancing mechanism of the mechanical properties by using compressive residual stresses in the matrix of Al/TiNi composites. After TiNi wires of austenite phase were composed in Al matrix, the composites were quenched. The as-fabricated composite is aged at shape-memorizing temperature and then the composite is cooled to martensite phase. When this composite of martensite phase is subjected to tensile prestrain and heated above austenite finish temperature(A_f), TiNi wire shrinks to the original length by shape memory effect. Thus this shrinkage induces the compressive residual stress in Al matrix along the fiber axis while the fiber is in tension.

Al/TiNi composite can be fabricated by powder metallurgy or casting method. In this study, Al/TiNi composite was fabricated by squeeze casting method [6],[7]. To get the optimum condition in fabrication of squeeze cast Al/TiNi composite, processing factors such as the temperature of molten Al, preheating temperature of TiNi wire and mold, applied pressure, and duration time were considered. The interface between Al matrix and TiNi reinforcement of fabricated SMC was analyzed using SEM and EDX. Tensile tests of TiNi wire and the composite were carried out to characterize the shape memory effect in SMC.

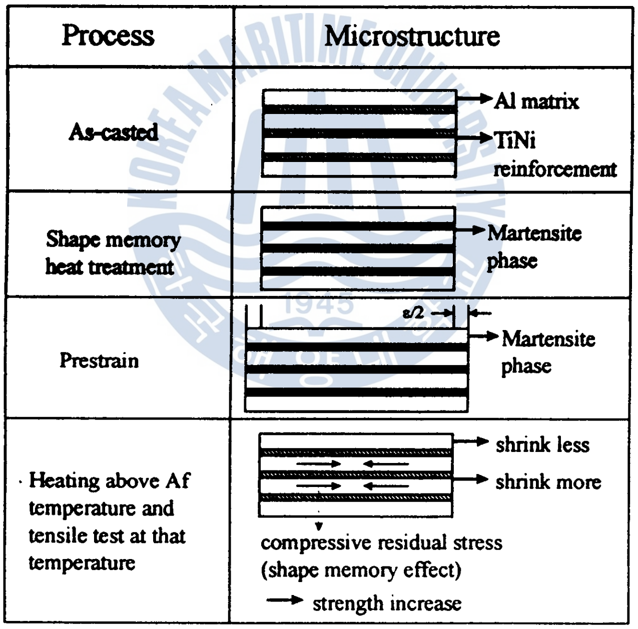


Fig. 1 Enhancing mechanism of SMC

EXPERIMENTAL

Fabrication of Al/TiNi shape memory composites

AC4A Al was chosen as matrix because of their good castability, strength and corrosion resistance under high pressure. Diameter of 0.38 and 1mm TiNi wire were chosen as reinforcement because of their large shape memory effect and high stiffness. First of all, the interfacial bonding strength of Al/TiNi composites was investigated as a fundamental study in order to decide manufacturing conditions of composites. The squeeze casting was performed with a range of pouring temperature of 700~900℃ and mold preheating temperature of 300~500℃. The applied pressure was fixed at 75MPa. The pull-out strength of fabricated composites was obtained through pull-out test designed as shown in Figure 2. The surface condition of TiNi wire affected the interfacial property. The specimen with acid cleaned surface had higher pull-out strength, but the wire without acid cleaning pulled out easily. However, the interfacial bond strength of Al/TiNi didn't change much according to the conditions of pouring temperature and mold preheating temperature. Therefore, in this study, the wire was cleaned in acid solution to have good wettability between matrix and reinforcement and the squeeze casting conditions were set as 400℃ of mold preheating temperature and 750℃ of pouring temperature of molten Al in considering infiltration velocity of Al melt, matrix microstructure and preform deformation.

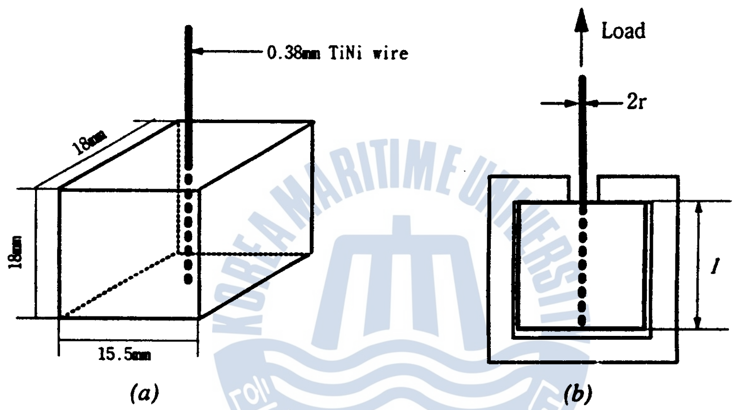


Fig. 2 (a) Pull-out test specimen, (b) Loading for pull-out test

The preform was woven by TiNi wire into the perforated jig(sus.304) as shown in figure 3. The volume fraction of reinforcement was 5-7%. To get off the oxidation film on TiNi wire, the preform was acid cleaned for 120-180 second in 16% HNO_3 + 1%HF bath. Squeeze casting was carried out by pouring molten Al of 750℃ into the TiNi preform, which was placed in the mold preheated to 400℃, and then by applying pressure of 75MPa. In applying pressure, delay time and duration time were 7 and 60 seconds, respectively. The processing chart is shown in figure 4.

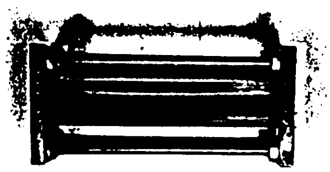


Fig. 3 Preform fabricated with 0.38mm TiNi wire

The direction of applied pressure was perpendicular to longitudinal direction of TiNi wire. The squeeze cast block was quenched into water after applying pressure and the composite block fabricated through this process is shown in figure 5(a). Squeeze cast Al/TiNi composites were tested by X-ray to confirm the straightness of TiNi wire in composites(figure 5(b)). Composites with bending deformation of TiNi wire were discarded .

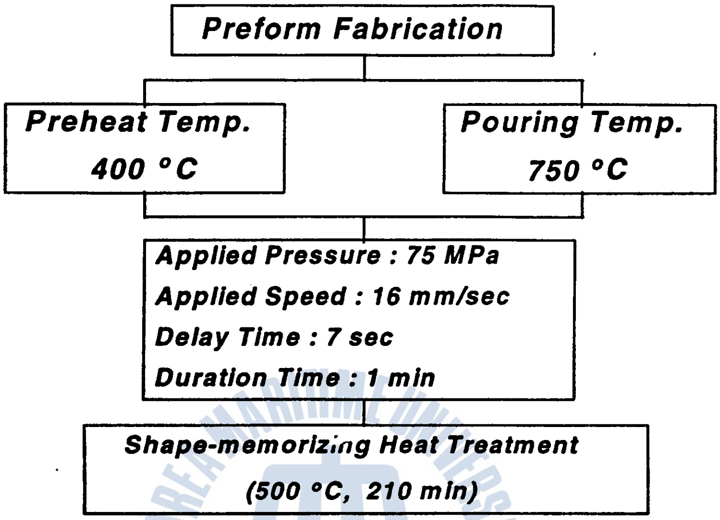


Fig. 4 Flowchart for Al/TiNi SMC manufacturing process by squeeze casting method

Fig. 5 (a) Squeeze cast shape memory composite fabricated
(b) Non-destructive test by X-ray

Microstructure and tensile test

Solidification microstructure of composites was examined and analyzed with optical microscope and SEM(JEOL JSM-5400) and the diffusion distance of Al/TiNi interface was checked with EDX analysis.

Transformation temperatures of TiNi wire which was aged at 500℃ for 210 minutes followed by water quenching to induce martensitic transformation of TiNi fiber were measured using differential scanning calorimeter with a heating rate 10℃/min to decide the temperature of tensile testing. The superelastic region caused by the stress-induced martensitic phases was identified by the tensile test of TiNi wire.

Tensile specimens of composites were machined by electro-discharge machine and the dimension of tensile specimen is shown in Figure 6. Each coupon was heat treated for shape memorizing at 500℃ for 210 minutes followed by water quenching to induce martensitic transformation. From the results of TiNi wire tensile test, composites with martensitic TiNi reinforcement were prestrained up to 4% elongation. Tensile tests were carried out at 90℃ (above A_f: austenite finish temperature) with strain rate of 1mm/min.

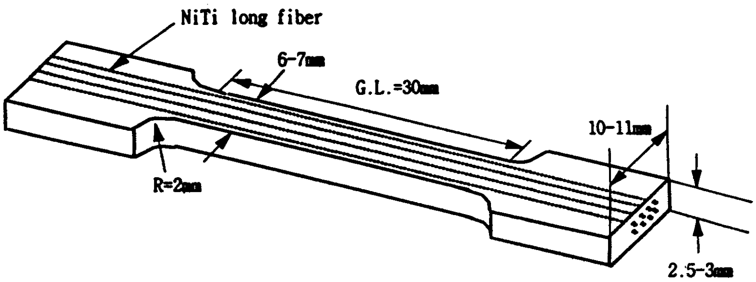


Fig. 6 Size of tensile test specimen.

RESULTS AND DISCUSSION

Microstructure

Figure 7 is the optical micrograph of the squeeze cast Al/TiNi shape memory composite. The marking of a and b in figure represent TiNi continuous fiber and AC4A matrix, respectively. Fine microstructure which had no cast defect in Al matrix and the matrix/reinforcement interface was obtained. Matrix is typical Al-Si cast microstructure that consists of eutectic structure around the primary α. It is considered that all TiNi did not have much role as nucleation sites because matrix/reinforcement interface mainly composed of eutectic structure with few primary α which is nucleated at high temperature.

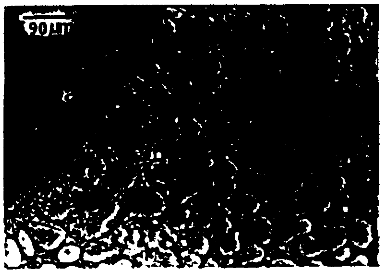


Fig. 7 Optical micrograph of AC4A Al / TiNi shape memory composite.
(a : TiNi reinforcement, b: Al matrix)

SEM-EDX analysis was carried out in order to identify the diffusion distance of each element at Al/TiNi interface. Figure 8 shows the interface of Al/TiNi composite which was infiltrated by 75MPa at optimum condition(750℃ of pouring temperature, 400℃ of mold preheat temperature) and was subjected to heat treatment at 500℃ for 210 minutes to shape-memorize TiNi wire. It was estimated that Al was diffused by 1.09㎛ from Al to TiNi and this specimen showed fairly good pull-out strength. Al/TiNi composites with small diffusion distance fabricated at other conditions exhibited easy pull-out of TiNi wire from matrix.

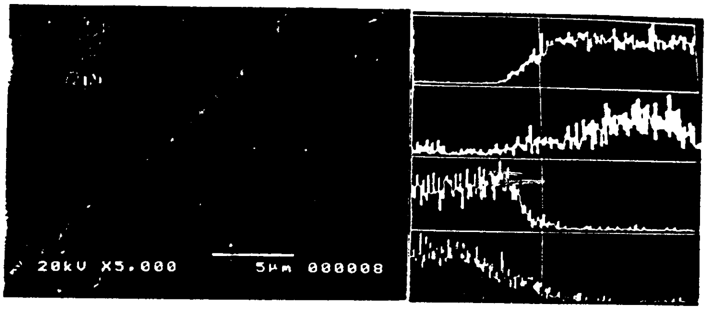


Fig. 8 SEM microstructure and EDX analysis of the interfaces of shape memory composite aged at 500 ℃ for 210 min to shape-memorize. (X_{Al} = 1.09 : Diffusion distance from Al into TiNi)

Tensile Properties of TiNi wire

The transformation temperature(martensite-austenite) and tensile property of shape memory alloy have been reported to change with the condition of shape-memorizing heat treatment[8],[9],[10]. Accordingly, tensile and transformation behavior of TiNi wire must be ascertained in advance to evaluate tensile properties of squeeze cast Al/TiNi composites.

Austenite finish temperature of TiNi wire aged to shape-memorize at 500℃ for 210 minutes was 57.7℃ from the result of DSC(differential scanning calorimetry) analysis. The composite was subjected to tensile prestrain when TiNi wire was in martensitic phase and then heated above A_f. The tensile test of composite at this temperature showed the effect of compressive residual stress due to the shape recovery by the austenite transformation of TiNi in Al matrix. The prestrain and the shape recovery force can be deduced from the tensile test of TiNi wire as shown in figure 9. At room temperature, TiNi wire exhibits the superelastic region(flat region) of 7-8% due to stress-induced martensite. The superelastic region is appeared in tensile curve of TiNi wire tested at room temperature and 50℃, but this region is not shown at 90℃. This fact is useful in determining the temperatures at which prestrain is to be made, and also at which tensile testing of the composite specimen is to be conducted. Accordingly, in this study, the prestrain was determined as 4% at room temperature and tensile test was carried out at 90℃. It can be predicted that shape recovery force of TiNi wire is about 250MPa if wire is prestrained at room temperature and tested at 90℃.

Tensile properties of Al/TiNi composite.

Figure 10(a) is the result of tensile test of squeeze cast Al/TiNi composite. The volume fraction of TiNi reinforcement was 5.5% and 6.6% in as-fabricated composite. Al/6.6%TiNi composite without prestrain exhibited the strength increment of 50MPa by fiber strengthening in comparison with that of Al matrix at the test temperature of 90℃. The strength of Al/6.6%TiNi composite with 4% prestrain increased with 45MPa in comparison with that of 0% prestrain. This was caused by the compressive residual stress due to the shape recovery force of TiNi reinforcement. The strength increment was also shown in Al/5.5%TiNi composite. But this increment was smaller than that of Al/6.6%TiNi composite. Thus the strength increment is increased with increasing the volume fraction of TiNi reinforcement.

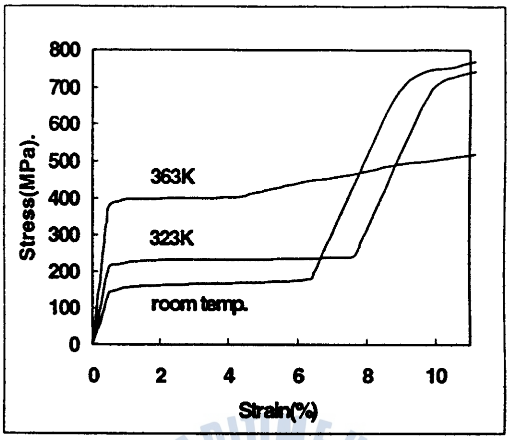
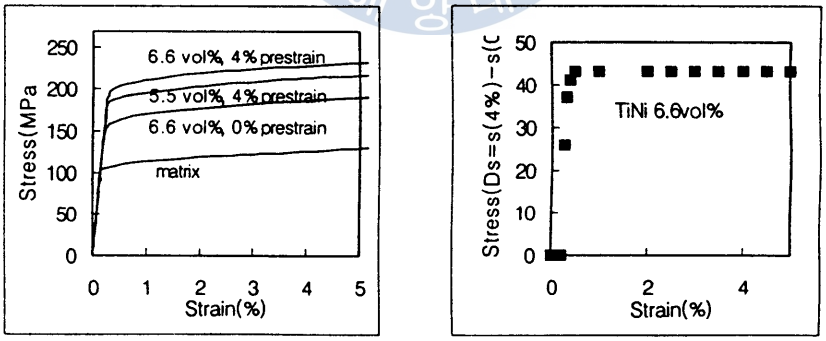


Fig. 9 Tensile curves of TiNi wire heat treated at 500 ℃ for 210 min.

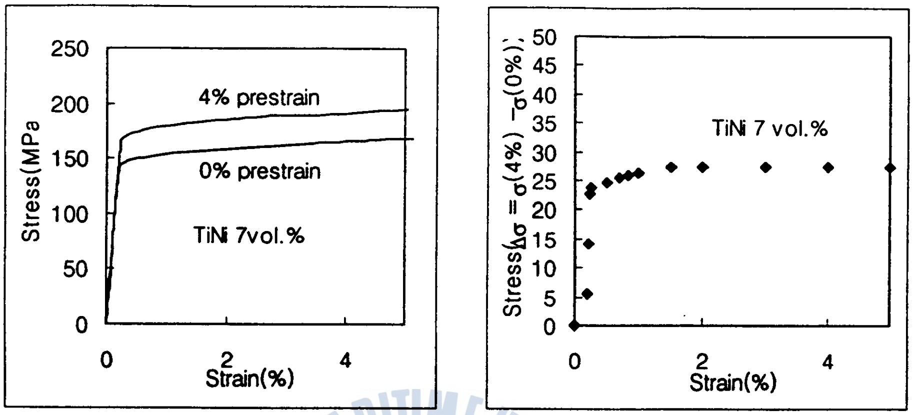
The strength increment by shape recovery force with the strain in Al/6.6%TiNi is shown in figure 10(b). The strength is increasing gradually up to 1% strain, thereafter the strength increment is constant as 45MPa. When the yield strength of TiNi wire and Al matrix are 400MPa and 100MPa, respectively, from tensile curves of figure 9 and figure 10(a), the experimental data of composite yield strength have higher value than theoretical value predicted by rule of mixture. But the discrepancy between experimental and theoretical value will be decreased with considering the additional strength increment and the back stress which are caused by TiNi strengthening with the plastic deformation of Al matrix.



(a) (b)

Fig. 10 The result of tensile test of the composite reinforced by 0.38mm TiNi

Figure 11 shows the result of tensile test of composite reinforced with 1mm TiNi wire. The volume fraction of reinforcement is 7%. The strength of this composite without prestrain is nearly the same as that of the composite reinforced with 0.38mm TiNi wire. The strength of composite with 4% prestrain increased with 20MPa in comparison with that of 0% prestrain. The compressive residual stress due to the shape recovery force of TiNi reinforcement is also identified in Al/1mm TiNi composite. However, the strength increment of this composite is smaller than that of the Al/0.38mmTiNi composite. This is caused by the difference of Al/TiNi interfacial area. The interfacial area of Al/0.38mmTiNi composite is about twice as large as that of Al/1mmTiNi composite. Therefore, the effect of compressive residual stress



(a) *(b)*

Fig. 11 The result of tensile test of the composite reinforced by 1mm TiNi.

due to the shape recovery force of TiNi on the strength of SMC is dependent on the area of Al/TiNi interface, that is, the strength of SMC is increased with decreasing the diameter of TiNi wire under condition of the same volume fraction of reinforcement. Even if there is an analysis only as a function of the volume fraction of reinforcement [11], no analytical study for the effect of the interfacial area has been performed yet for the strengthening mechanism of SMC. Therefore, more systematic theoretical analysis is needed to confirm the strengthening of SMC due to the interfacial area.

CONCLUSION

The optimum condition to fabricate Al/TiNi composite by squeeze casting was deduced. The composite fabricated at this condition had good interfacial bonding between Al matrix and TiNi reinforcement. In order to improve the interfacial bonding, TiNi wire was cleaned in acid solution. Transformation and tensile behaviors of TiNi wire was investigated to characterize the tensile property of the squeeze cast Al/TiNi composite. The composite was subjected to 4% prestrain at room temperature to induce compressive residual stress and then tensile test of the composite was carried out at 90℃. Improvement of strength was obtained by 50MPa and 45MPa in Al/0.38mmTiNi composite due to TiNi fiber strengthening and induced compressive residual stress, respectively. However, the strength increment due to compressive residual stress of Al/1mmTiNi composite was 20MPa. This difference of strength increment is caused by that of Al/TiNi interfacial area. Therefore, the effect of compressive residual stress due to the shape recovery force of TiNi on the strength of SMC is increased with decreasing the diameter of TiNi wire.

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