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Effect of high pressure during the fabrication on the thermal and mechanical properties of amorphous $Ni_{60}Nb_{40}$ particle-reinforced Al-based metal matrix composites

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$Ni_{60}Nb_{40}$ metallic glass particles, prepared by mechanical alloying, were used to fabricate Al–30 wt% $Ni_{60}Nb_{40}$ metal matrix composites (MMCs) at 823 K by sintering, hot pressing, and hot extrusion. The $Ni_{60}Nb_{40}$ reinforcements remained amorphous in all the MMCs fabricated by the different methods. Compression tests revealed that the Young's modulus and yield strength of the MMCs produced by hot-pressing and hot-extrusion are higher than those for the sintered specimens. Differential scanning calorimeter (DSC) annealing of the as-produced MMCs at 913 K revealed there are severe interfacial reactions between the Al matrix and the $Ni_{60}Nb_{40}$ reinforcements. The DSC isotherms indicate that the Al matrix reacts faster with $Ni_{60}Nb_{40}$ in the hot-pressed and hot-extruded MMCs than it does in the sintered material.

I. INTRODUCTION

Metallic glasses have been drawing increasing attention in recent years due to their scientific and engineering significance.[1–3] Compared to conventional metals and alloys, metallic glasses have superior properties such as high strength, hardness, and elastic strain limit, along with relatively high fracture toughness, fatigue, and corrosion resistance.[4] Therefore, metallic glasses are considered promising structural materials[5,6] and have already found industrial applications, such as golf clubs, mobile phone casings, and surgical instruments.[7]

Recently, there have been some attempts to use metallic glass as reinforcement in metal matrix composites (MMCs).[8–10] It was believed that by replacing conventional ceramic reinforcements with metallic glass, the matrix-reinforcement interfacial bonding can be improved, which should lead to better mechanical properties.[8,10] The first attempt was made by Lee et al.[8,9] In their work, an infiltration casting method was used to produce Al-based matrix composites in which Ni–Nb–Ta metallic glass, in the form of either ribbons or particles, served as reinforcements.[8,9] These composites exhibit higher strength than their monolithic matrix alloys.

The most important factor in fabrication of metallic glass-reinforced MMCs is to prevent the metallic glass reinforcement from crystallizing or reacting with the matrix, so the highest temperature during fabrication should be lower than the crystallization temperature of the selected glass. Considering that the crystallization temperatures for most of the available metallic glasses are typically lower than the melting temperatures of Al and Mg,[11] two of the most commonly used matrix materials for MMCs,[12] it is necessary to develop some techniques by which metallic glass-reinforced metal matrix composites can be produced at temperatures below the melting temperature of the matrix. In our previous work, we produced Ni–Nb metallic glass particle-reinforced Al-based MMCs by sintering at a temperature lower than the melting temperature of Al.[10] Mechanical testing showed improved strength and Young's modulus compared to the matrix material.

Hot-pressing and hot-extrusion are other widely used techniques for fabricating conventional Al-based MMCs at temperatures lower than the melting temperature of the Al matrix.[12] However, there is still no report of application of hot pressing and hot extrusion to the fabrication of metallic glass-reinforced MMCs. Moreover, although

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previous investigations indicate that high pressure has an important influence on the formation and crystallization processes of metallic glasses,[13,14] there has been so far no study that investigates the effect of high pressure during fabrication on the microstructures and properties of metallic glass-reinforced MMCs.

In this paper, $Ni_{60}Nb_{40}$ metallic glass powder is prepared by mechanical alloying and is then used to fabricate Al–30 wt% $Ni_{60}Nb_{40}$ MMCs by three different methods, i.e., sintering, hot pressing, and hot extrusion. The microstructures, as well as the thermal and mechanical properties of the MMCs prepared by the different methods, were investigated and compared.

II. EXPERIMENTAL PROCEDURES

Pure Ni (99.8%, 2 μm) and Nb (99.8%, 2 μm) powders were used to prepare metallic glass reinforcement particles with a nominal composition of $Ni_{60}Ni_{40}$. The mixture of Ni and Nb powders was put into a steel vial together with some stainless steel balls. The ball-to-powder ratio was 10:1. The vials were sealed in a glove box in an Ar atmosphere. A Fritsch Pulverisette 5 planetary ball mill (Idar-Oberstein, Germany) was used to mill the powders for 20 h at a rotational speed of 200 rmp.

In a second step, pure Al powder (99.5%, 325 mesh) was mixed with 30 wt% as-milled $Ni_{60}Nb_{40}$ glassy powder in a mortar, and then some of the mixed powder was cold-pressed under 400 MPa to form green compact disks 10 mm in diameter. The Al–30 wt% $Ni_{60}Nb_{40}$ green compacts were sintered in a tube furnace for 1800 s, and flowing Ar was used to prevent the samples from oxidation. According to the results from the previous differential scanning calorimeter (DSC) study,[15,16] the sintering temperature was fixed at 823 K. Other green compacts were subject to hot extrusion. They were heated to 823 K for 1800 s in vacuum and then extruded with an extrusion ratio of 10:3 to form rods 3 mm in diameter. In addition, Al–30 wt% $Ni_{60}Nb_{40}$ powders were put into a hot press, heated to 823 K, and pressed under 400 MPa for 1800 s in a vacuum.

The density of the MMC samples prepared by sintering, hot pressing, and hot extrusion was measured by the Archimedian method. A Siemens D500 (Karlsruhe, Germany) x-ray diffractometer (XRD; Cu K_α radiation) was used for phase identification. A Zeiss DSM 962 (Oberkochen, Germany) scanning electron microscope (SEM) equipped with a Noran (Waltham, MA) energy-dispersive x-ray analysis (EDX) system was used for microstructure observation and composition analysis. To measure the mechanical properties, bar-shape specimens with a dimension of 4 mm × 2 mm × 2 mm were machined from the sintered, hot pressed, and hotextruded MMC samples, and compression tests were conducted on these specimens in a Schenck (Darmstadt, Germany) hydraulic testing machine under quasistatic loading at an initial strain rate of $8 \times 10^{-4}\ s^{-1}$ at room temperature. To investigate the thermal properties of the samples prepared by different methods, the samples were isothermally annealed at 913 K in a Perkin-Elmer Diamond (Shelton, CT) DSC for 4000 s. The as-annealed samples were also investigated by XRD and SEM.

III. EXPERIMENTAL RESULTS

Figure 1(a) shows a SEM micrograph of 20 h milled $Ni_{60}Nb_{40}$ powder. The milled powder particles are equiaxied. The particle size varies from 50 to 100 μm. The EDX results reveal that the as-milled powder contains 59.8 at.% Ni, 38.6 at.% Nb, and 1.6 at.% Fe. The composition is close to the desired composition. The iron content in the milled powders comes from the debris of the vials and balls during milling. The SEM micrographs of the Al–30 wt% $Ni_{60}Nb_{40}$ MMC samples prepared by sintering, hot pressing, and hot extrusion are shown in Figs. 1(b), 1(c), and 1(d), respectively. Bright $Ni_{60}Nb_{40}$ particles are distributed randomly in the dark Al matrix. The interface between the Al matrix and the $Ni_{60}Nb_{40}$ metallic glass reinforcement is clean, and no reaction zone is seen along the interface.

XRD was used to check the structure of the as-milled $Ni_{60}Nb_{40}$ powder as well as that of the Al–30 wt% $Ni_{60}Nb_{40}$ MMC samples prepared by sintering, hot pressing, and hot extrusion. The XRD patterns are shown in Fig. 2. The XRD pattern of as-milled $Ni_{60}Nb_{40}$ displays only broad diffuse diffraction maxima corresponding to an amorphous structure. The amorphous state of the mechanically alloyed powder was also proved by transmission electron microscopy (see Ref. 10). The three XRD patterns of the sintered, hot pressed, and hot extruded Al–30 wt% $Ni_{60}Nb_{40}$ MMCs are almost identical: all exhibit sharp crystalline peaks corresponding to pure Al on top of the maxima of the $Ni_{60}Nb_{40}$ metallic glass.

The densities for the sintered, hot pressed, and hot extruded Al 30 wt% $Ni_{60}Nb_{40}$ MMCs are 3.026, 3.259, and 3.360 g/cm^3 (Table I). The theoretical density of the Al–30 wt% $Ni_{60}Nb_{40}$ MMC is determined to be 3.405 g/cm^3, according to the densities and the weight fractions of each component in the MMC. Accordingly, the relative densities for the differently prepared MMCs were calculated, and the values are also shown in Table I. They are 88.9% (sintered), 95.7% (hot-pressed), and 98.7% (hot-extruded).

According to our previous investigations,[16] the crystallization temperature for $Ni_{60}Nb_{40}$ is 925 K, and interfacial reactions between the Al matrix and the $Ni_{60}Nb_{40}$ reinforcement are observed when the MMCs are subjected to lengthy DSC annealing at temperatures slightly

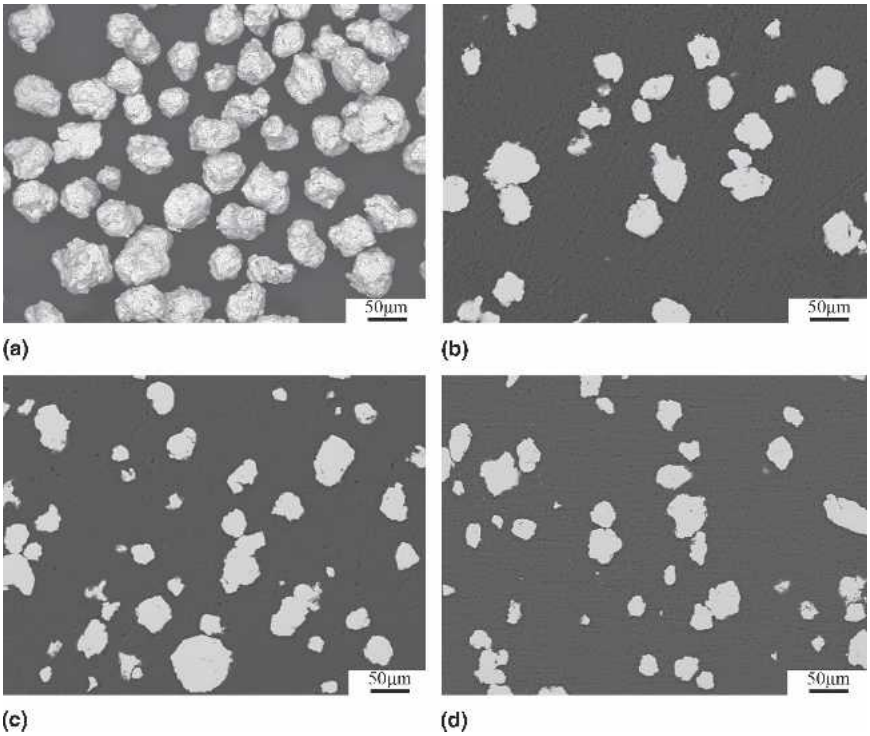

FIG. 1. SEM micrographs of (a) $Ni_{60}Nb_{40}$ metallic glass particles fabricated by mechanical alloying of Ni and Nb powders for 7200 s and of the Al-30 wt% $Ni_{60}Nb_{40}$ MMCs prepared at 823 K by (b) sintering, (c) hot-pressing, and (d) hot-extrusion.

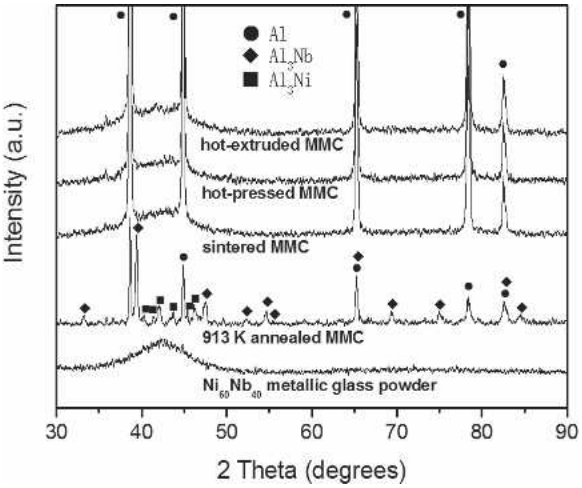

FIG. 2. XRD patterns of $Ni_{60}Nb_{40}$ metallic glass particles fabricated by mechanical alloying of Ni and Nb powders for 7200 s and of the Al–30 wt% $Ni_{60}Nb_{40}$ MMCs prepared at 823 K by sintering, hot pressing, and hot extrusion, and of the Al–30 wt% $Ni_{60}Nb_{40}$ MMCs annealed at 913 K for 4000 s.

TABLE I. Summary of the densities, relative densities, Young's moduli, and yield strengths, as well as the onset times, end times, and interfacial reaction heat for Al–30 wt% $Ni_{60}Nb_{40}$ MMCs prepared at 823 K by sintering, hot pressing, and hot extrusion.

	Furnace sintering	Hot pressing	Hot extrusion
Density (g/cm^3)	3.026	3.259	3.360
Relative density (%)	88.9	95.7	98.7
Onset time of the interfacial reaction (s)	1400	860	820
End time of the interfacial reactions (s)	2700	2100	2000
Heat released in the interfacial reaction (J/g)	546	484	476
Young's modulus E (GPa)	65	67	72
Yield strength $\sigma_{0.02}$ (MPa)	94	106	134

lower than the crystallization temperature of $Ni_{60}Nb_{40}$. Investigations of the interfacial reactions in Al–30 wt% $Ni_{60}Nb_{40}$ MMCs prepared by different methods can give information about the effect of high pressure on the thermal properties of the consolidated MMCs. Therefore, three MMCs that were prepared at 823 K by sintering, hot pressing, and hot extrusion were isothermally annealed in the DSC at 913 K, a temperature slightly lower than the crystallization temperature of $Ni_{60}Nb_{40}$. The corresponding DSC isotherms are shown in Fig. 3. Each isotherm exhibits an exothermic peak representing the interfacial reaction. The exothermic peaks for the hot-pressed and hot extruded MMCs appear at shorter times compared with the signal from the sintered MMC. The onset time and the end time of the exothermic peaks for the three samples are summarized in Table I. The onset times for the sintered, hot pressed, and hot extruded Al–30 wt% $Ni_{60}Nb_{40}$ MMCs are 1400, 860, and 820 s, while the end times are 2700, 2100, and 2000 s, respectively. Both times demonstrate that the exotherms appear earlier as the relative density of the samples increases. The heat

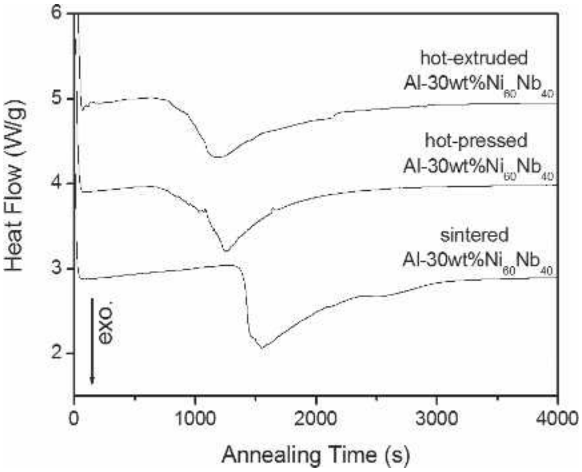

FIG. 3. DSC isotherms of the sintered, hot pressed, and hot extruded Al–30 wt% $Ni_{60}Nb_{40}$ MMCs annealed at 913 K for 4000 s.

flow corresponding to the exothermic peaks for the sintered, hot pressed, and hot extruded Al–30 wt% $Ni_{60}Nb_{40}$ MMCs was also calculated and is listed in Table I. The values are 546, 484, and 476 J/g, respectively, indicating that the released heat decreases with increasing relative density.

To understand the microstructures of the MMC samples after annealing, XRD and SEM were used to investigate the sintered Al–30 wt% $Ni_{60}Nb_{40}$ MMC after 913 K DSC annealing. A typical XRD pattern of the 913 K annealed Al–30 wt% $Ni_{60}Nb_{40}$ MMCs is shown in Fig. 2. The intensity maximum representing the amorphous $Ni_{60}Nb_{40}$ reinforcements disappears, and some peaks corresponding to Al_3Ni and Al_3Nb intermetallics are found. Figure 4 shows a SEM micrograph of the sintered Al–30 wt% $Ni_{60}Nb_{40}$ MMC after 913 K DSC annealing. The amount of Al matrix is much smaller in the case of the unsintered samples, and the initial glassy $Ni_{60}Nb_{40}$ reinforcements are replaced by some 2-layer structural particles. EDX investigation shows that the inner reaction layer contains 74.5 at.% Al, 0.5 at.% Ni, and 25.0 at.% Nb, while the outer layer has a composition of 73.3 at.% Al, 23.3 at.% Ni, and 3.4 at.% Nb, which suggests that the inner layer is Al_3Nb and the outer layer Al_3Ni.

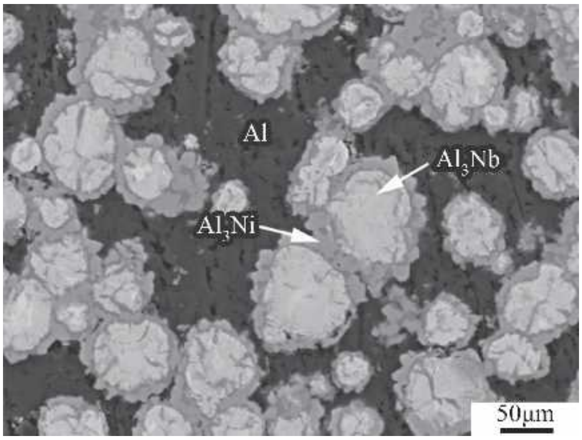

FIG. 4. SEM micrograph of the hot-extruded Al–30 wt% $Ni_{60}Nb_{40}$ MMC after DSC annealing at 913 K for 4000 s.

Compression tests were conducted for the sintered, hot pressed, and hot extruded Al–30 wt% $Ni_{60}Nb_{40}$ MMC samples. The obtained stress–strain curves are shown in Fig. 5, and the results are also summarized in Table I. The Young's modulus and yield strength are increased by the high pressure used upon hot pressing and hot extrusion. The Young's modulus for the sintered, hot pressed, and hot extruded Al–30 wt% $Ni_{60}Nb_{40}$ MMCs are 65, 67, and 72 GPa, and their yield strengths are 94, 106, and 134 MPa, respectively.

IV. DISCUSSION

Al–30 wt% $Ni_{60}Nb_{40}$ MMC samples have been prepared at the same temperature of 823 K by three different methods, i.e., sintering, hot pressing, and hot extrusion. For all the differently prepared MMCs, the $Ni_{60}Nb_{40}$ metallic glass reinforcements survive the high temperature upon fabrication and maintain their amorphous state. However, it seems that the pressure used in the fabrication has a great influence on the properties of the final products.

On one hand, high pressure strongly increases the relative density of the MMCs. Accordingly, the resulting mechanical properties for the hot-pressed and the hot-extruded Al–30 wt% $Ni_{60}Nb_{40}$ MMCs are also improved due to the better matrix-reinforcement interfacial bonding in the MMC fabricated under high pressure. The Young's modulus and the yield strength of the hot-pressed Al–30 wt% $Ni_{60}Nb_{40}$ MMC are 3.2% and 12.8% higher than for the sintered MMC. The corresponding

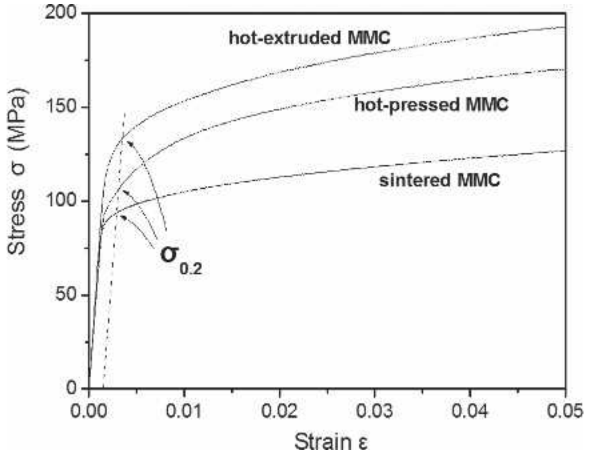

FIG. 5. Compression test stress–strain curves for the Al–30 wt% $Ni_{60}Nb_{40}$ MMCs prepared at 823 K by sintering, hot pressing, and hot extrusion.

values for the hot-extruded Al–30 wt% $Ni_{60}Nb_{40}$ MMC are 10.7% and 42.6% higher than that for the sintered MMC. In this sense, hot pressing and hot extrusion are better methods than sintering for the fabrication of metallic glass reinforced MMCs.

On the other hand, the high pressure also affects the thermal properties of the MMC. The DSC isotherms of the three Al–30 wt% $Ni_{60}Nb_{40}$ MMCs in Fig. 3, and the SEM micrograph in Fig. 4 show that there are severe matrix-reinforcement interfacial reactions between the Al matrix and the $Ni_{60}Nb_{40}$ reinforcements at 913 K. During the interfacial reactions, the Al atoms in the matrix diffuse into the $Ni_{60}Nb_{40}$ reinforcement to react with Nb to form Al_3Nb intermetallic; at the same time, the Ni atoms diffuse out of the $Ni_{60}Nb_{40}$ reinforcements to react with Al matrix to form Al_3Ni intermetallic. The resulting reaction product is a two-layer intermetallic structure found in Fig. 4, and the overall reaction is shown as below:

$$Al_{(s)} + Ni_{60}Nb_{40(s)} \rightarrow Al_3Ni_{(s)} + Al_3Nb_{(s)} \quad . \qquad (1)$$

The DSC curves in Fig. 3 indicate that the interfacial reactions for the hot-pressed and hot-extruded MMCs occur earlier and the heat release during the reaction becomes smaller than that for the sintered MMC. Two factors are believed to be responsible for the differences. First, the better matrix-reinforcement interfacial bonding in the hot-pressed and hot-extruded MMCs makes the necessary mass transfer across the matrix-reinforcement boundary during the interfacial reactions easier than in the sintered MMC. Second, our previous research[16] and the work of Lee et al.[8] demonstrate that interfacial reactions in the Ni–Nb-based metallic glass-reinforced Al-based MMCs are to a great extent controlled by the structure of the Ni–Nb-based reinforcements. When the Ni–Nb-based reinforcements are amorphous, the reaction rate for the matrix-reinforcement reactions is extremely low, but the matrix-reinforcement reactions strongly speed up immediately after the Ni–Nb-based reinforcements are crystallized.[16] At the same time, it is widely recognized that the crystallization of metallic glasses is sensitive to the applied pressure.[17] Many investigations[18,19] have shown evidence that high pressure promotes short-range rearrangements of the atoms before crystallization of the metallic glasses, since pressure can lead to annihilation of free volume, reduces voids through compressing the glassy structure, and reconstructs the atomic configuration. These effects are related to the short-range rearrangement of atoms. The rearrangement favors the homogeneous formation of small clusters, which distribute uniformly in the amorphous matrix and can act as nucleation sites.[18,19] It was also reported that, because of the above reasons, high temperature promotes the relaxation and the crystallization processes of metallic glasses,[20] and their glass transition temperatures and crystallization temperatures are reduced under high pressure.[17] According to the above findings, it is reasonable to assume that in our case, the high pressure in hot-pressing and hot-extrusion changes the short-range order of the $Ni_{60}Nb_{40}$ glassy reinforcements and introduces clusters. As a result, the pressure-introduced clusters promote the crystallization of the $Ni_{60}Nb_{40}$ glassy reinforcements and speed up the matrix-reinforcement reaction during the subsequent isothermal DSC annealing. Therefore, the high pressure during the Al–30 wt% $Ni_{60}Nb_{40}$ MMC fabrication decreases the stability of the matrix-reinforcement interface, which makes the $Ni_{60}Nb_{40}$ reinforcement more prone to degeneration and loss of its special mechanical properties, such as high hardness and high strength, especially when the MMC serves at high temperatures.

The DSC results indicate that the interfacial reaction heat for the hot-pressing and hot-extruded samples is less than that for the sintered sample. Because the SEM micrographs in Fig. 1 show all the MMCs prepared by the three different methods have clear reinforcement-matrix interfaces, indicating no interfacial reactions during sample preparation at 823 K, the difference of reaction heat can be attributed only to the relaxation of $Ni_{60}Nb_{40}$ reinforcements while they are subject to high pressure. It is well known that the enthalpy of the metallic glass decreases irreversibly upon heating for the first time due to relaxation.[21,22] Therefore, when high pressure promotes the relaxation of $Ni_{60}Nb_{40}$ reinforcement during the MMC preparation, it also helps to decrease the enthalpy of the $Ni_{60}Nb_{40}$ reinforcement and that of the whole MMC at the same time. As a result, when the MMCs are subject to higher temperature, i.e., 913 K, and the interfacial reactions occur, the enthalpy of the reactants for hot-pressed and hot-extruded MMCs is lower than that for the sintered one, which leads to different reaction heat for three MMCs prepared by different methods in reaction (1).

V. CONCLUSIONS

$Ni_{60}Nb_{40}$ metallic glass particles were prepared by mechanical alloying and were used to fabricate Al–30 wt% $Ni_{60}Nb_{40}$ MMCs at 823 K by sintering, hot pressing, and hot extrusion, respectively. The glassy $Ni_{60}Nb_{40}$ reinforcements survive the high temperatures during fabrication and remain amorphous in the final MMC products, showing a clear interface with the Al matrix. The pressure during the fabrication increases not only the relative density but also the Young's modulus and the yield strength of the MMC. Severe interfacial reactions occur when the MMCs were subsequently isothermally annealed at 913 K in the DSC. The DSC isotherms reveal that the high pressure during the fabrication deteriorates the matrix-reinforcement stability of the MMC and speeds up the interfacial reactions between the Al matrix and the $Ni_{60}Nb_{40}$ reinforcements during annealing.

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