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A Hot Extrusion Process without Sintering by Applying MWCNTs/Al6061 Composites

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Abstract: For carbon nanotube (CNT)/Al composites, compaction forming is conducted for densification processing, and then sintering and secondary processes are conducted. This general process has problems such as the complexity of the processing procedures, and high manufacturing costs. This study presents a hot extrusion process without sintering for fabrication of CNTs/Al6061 composites. Before hot extrusion, preforms are fabricated by the compaction process for the mixture of Al6061 powder and CNTs. Several hot extrusion experiments were performed under six types of CNT content; three extrusion ratios and three extrusion temperatures. The formability increased as the extrusion temperature increased for low CNT content. At 620 °C, the forming of all materials except for 10 vol % CNTs/Al6061 was possible at extrusion ratios $R = 4$, $R = 8$, and $R = 16$. As CNT content increases, extrusion pressure almost linearly increases. As the extrusion ratio increases, the extrusion pressure increases. The amount of CNT content increases as Vickers hardness increases. The Vicker's hardness of 1 vol % CNTs/Al6061 billet is about 100 HV while that of 10 vol % CNTs/Al6061 billet is about 230 HV. There are no significant differences of compression stress according to extrusion ratio as observed in terms of pure Al6061, 1 vol % CNT/Al6061, and 3 vol % CNTs/Al6061.

Keywords: carbon nanotubes; non-sintering; hot extrusion; aluminum 6061; extrusion ratio

1. Introduction

Recently, the development and application of lightweight materials has gained attention in the aerospace and automotive industries owing to their low energy consumption and reduced weight. Moreover, pollution can be decreased by using lightweight materials. As for the materials for weight lightening, the use of metals and nonmetallic materials is demanded.

Carbon nanotubes (CNTs) have been the subject of many investigations since CNTs were first discovered by Iijima in 1991 [1]. CNTs have generated a great deal of interest due to their unique properties of carbon structures with large aspect ratios and superior strength, stiffness, and thermal and electrical properties, together with their light weight [2–7]. CNTs are extensively used as reinforcement materials for several resin bases, ceramic bases, and metal matrix composites. The structure of a CNT is similar to the lamellar structure of graphite and, thus, CNTs are mainly divided into single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs). The C–C covalent bond by which carbon nanotubes are constituted is the most stable chemical bond in nature. CNTs have a higher modulus of elasticity or up to 1.8 TPa [8], and the strength of CNTs reaches up to 150 GPa [9].

The strength of CNTs is approximately 100 times higher than that of traditional steel, while the density of CNTs is 1/6 of that of the steel.

CNTs/Al6061 (purity > 95%) composites have further enhanced mechanical properties compared with CNTs/Pure Al (purity > 99.7%) [10]. CNTs/Al6061 composites manufactured using the powder compacting process showed higher strength than the raw materials, with a density higher than 80%. The material property of CNTs/Al6061 composites can be further enhanced with a densification process through hot extrusion [11].

Kuzumaki et al. [12] introduced a technology that can manufacture CNTs/Al composites using the hot compacting and hot extrusion processes. In the process of manufacturing CNTs/Al composites, not only was CNT damage within the Al matrix not observed, but carbides were also not generated when the thermal treatment was conducted for 24 h at a temperature of 983 K. Kim et al. [13] reported that the hardness and strength of CNTs/Al6061 composites were enhanced through hot compacting and hot extrusion, and that the mechanical properties were also enhanced according to compacting pressure and temperature variables. Zhou et al. [9] successfully conducted sintering at a temperature of 873 K using 5 vol % CNTs/Al6061 composite, as well as a hot extrusion experiment under the conditions of 823 K and 500 kN.

All the above-mentioned experiments explained the characteristics of the composite materials manufactured using the sintering process. In general, compaction forming is conducted for the densification process, and then the sintering and hot extrusion processes are conducted. If CNTs/Al6061 composites are manufactured using the above-mentioned methods, problems related to complex process procedures and high manufacturing costs may occur. As the hot extrusion process was conducted after the sintering process, the experiment procedures became complex, and the actual application in the industrial field becomes difficult. Because the investigation's results regarding the non-sintering of CNTs/Al6061 composites are inadequate, it is essential to investigate the variation in the mechanical properties of the CNTs/Al6061 composites under different conditions, such as extrusion temperature, extrusion ratio, and CNT content.

This study performed the hot extrusion process without sintering for fabrication of CNTs/Al6061 composites. Before hot extrusion, preforms are fabricated by the powder metallurgy forming process under different CNT contents. Hot extrusion billets from preforms are fabricated under certain extrusion temperatures and extrusion ratios. This non-sintering hot extrusion can be easily applied in the industrial field due to the simplified manufacturing procedure without the requirement to conduct a separate sintering process.

2. Experimental Methods

CNTs as a reinforcing agent were mixed with the matrix and embedded into Al6061 powder through high-energy ball milling (ACN, Seoul, Korea) (400 rpm, 30 min, methyl alcohol as PCA) [14]. CNTs were dispersed and mixed with Al6061 alloy at the ratio of 1 vol %, 3 vol %, 5 vol %, 7 vol %, and 10 vol %. Table 1 shows the chemical compositions of the CNTs and the Al6061 alloy.

Table 1. Properties of Al6061 alloy and multi-walled carbon nanotubes (MWCNTs).

Materials	Density (g/cm ³)	Mean Diameter (μm)	Tensile Strength (GPa)	Young's Modulus (GPa)	Melting Point (°C)
Al6061	2.70	5–30	0.12	63	582–652
MWCNTs	~1.8	0.02	100	1200	~3700

Figure 1 shows the compacting process for fabricating preforms. The compacting process was conducted at room temperature in the mode of ASTM Standard B925-08 [15]. The experiment was conducted in the pressure range between 400 MPa and 1000 MPa and the preliminary forming was performed with a pressure of 35 MPa. The compaction pressure was calculated as force/section area of die hole. In the experiment, the pressure was increased by 5.0 MPa/s until the required

compacting pressure was achieved. When the compacting time was 10 s, in accordance with the ASTM B331-95 Standard, the billet density of the specimen increased by 0.3% minimum [16]. The height and diameter of each billet were 19 mm and 12.7 mm, respectively. As the pressure reached the required compacting pressure, the pressure was removed for measuring the precise billet density. The billet density was measured using the Archimedes principle of ASTM B962-13 [17]. The relative densities versus compaction pressure are shown in Figure 2. Relative density of all conditions gradually increased with increasing compaction pressure. Relative density gradually decreased with increasing CNT content under the same compaction pressure. At the same compaction pressure, the relative density of Al6061 was more than that of the CNTs [18].

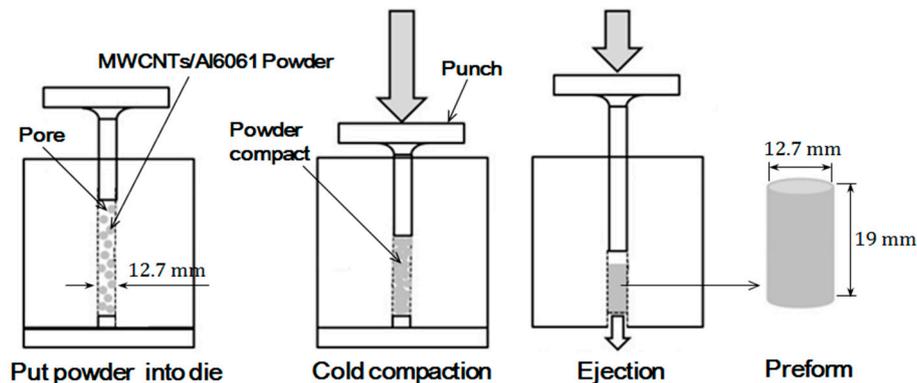


Figure 1. Schematic of the process for preform fabrication by cold-compaction process.

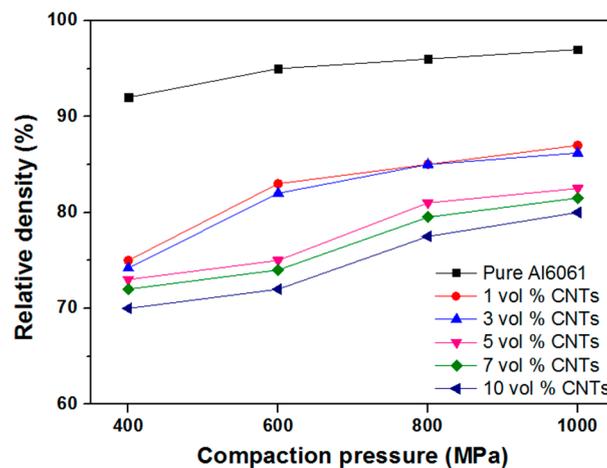


Figure 2. Relative density according to compaction pressure.

The hot extrusion process without sintering was conducted using the fabricated preforms. Figure 3 shows the schematic diagram of the hot extrusion tool. Extrusion angle is 45° and extrusion ratios are $R = 4$, $R = 8$, and $R = 16$, respectively. Extrusion ratio R is defined from Equation (1).

$$R = \frac{A_0}{A_1} \quad (1)$$

Here, A_0 is the cross-sectional area of the preform and A_1 is the cross-sectional area of the extrusion billet.

Extrusion experiments were conducted under different extrusion temperatures such as 520°C , 570°C , and 620°C . The extrusion speed is 2 mm/min. A total of 18 experiments were performed under six types of CNT content, three extrusion ratios, and three extrusion temperatures. Before

each extrusion experiment, graphite lubrication was coated at the die cavity. A preform was placed in an extrusion die and heated from the room temperature to the required temperature. When the temperature of the preform reached the required temperature, hot extrusion was performed by moving the punch. When the preform was pressurized, the punch recorded the displacement with load. When the preform passed through the extrusion die hole, the pressure was removed. Subsequently, the temperature was reduced to room temperature, and an extrusion specimen was removed from the die.

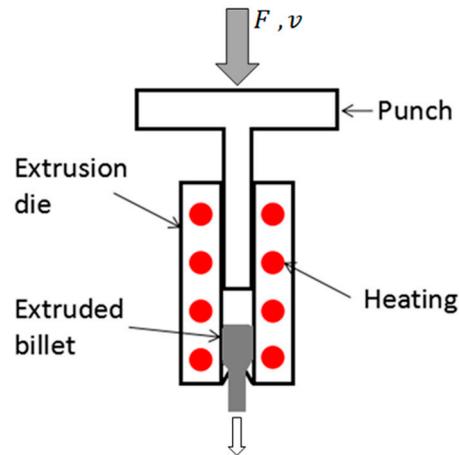


Figure 3. Schematic diagram of the hot extrusion process (F : extrusion force; v : velocity).

Figure 4 shows the position for microstructure determination and Vicker's hardness testing of the extrusion billets. The A position receives the highest pressure before the extrusion process, the B position undergoes the biggest deformation, and the C position follows the extrusion process. The load set for measuring Vickers hardness was fixed to 300 gf. The measurement of Vickers hardness was performed on the polished surface of the extrusion specimens. Scanning electron microscopy (SEM) (FEI Company, Hillsboro, OR, USA) was used to analyze the surface defects in the B position. The SE (secondary electron) mode was used for SEM. Metallographic examination (Olympus, Tokyo, Japan) was performed in the vertical directions of the specimens. The specimens were cut, mounted, ground, and polished. The specimens were etched for 10–12 s with a diluted solution composed of 190 mL H_2O , 5 mL HNO_3 , 3 mL HCL , and 2 HF. Thereafter, the microstructure, which represents the amount of Al (white phase) of each specimen, was captured using an optical microscope equipped with an image system.

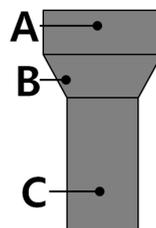


Figure 4. Three positions for microstructure and Vicker's hardness determination of extrusion billets.

3. Experimental Results and Discussion

The billets formed by the hot extrusion process are shown in Figure 5. Five vol % CNTs/Al6061 was perfectly formed at 570 °C extrusion temperature and an extrusion ratio of 4. This result is represented by the symbol "○". Seven vol % CNTs/Al6061 and 10 vol % CNTs/Al6061 was fractured during process. This result is represented by the symbol "x".

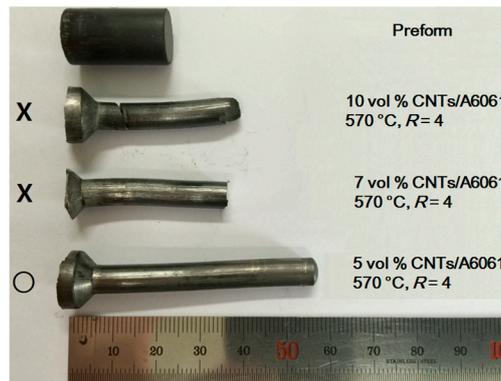


Figure 5. The preform and billets formed by the hot extrusion process.

In Figure 6, the possibility of hot extrusion forming was confirmed on the basis of parameters such as extrusion ratio, extrusion temperature, and CNT content. The formability increased as the extrusion temperature increased and the amount of CNT content is low. When the temperature of the billet was 520 °C, it was observed that Al6061—1 vol %, 3 vol %, and 5 vol %CNTs/Al6061—was formed under all extrusion ratios while the forming of 7 vol % and 10 vol % CNTs/Al6061 was impossible under all extrusion ratios. For 570 °C, the forming of 7 vol % CNTs/Al6061 was possible at extrusion ratios 8 and 16, but the forming was impossible at extrusion ratio 4. 10 vol % CNTs/Al6061 was not formed at any extrusion ratio. At 620 °C, the forming of all materials except for 10 vol % CNTs/Al6061 was possible at all extrusion ratios. 10 vol % CNTs/Al6061 was only possible at an extrusion ratio of 4. From the results of hot extrusion, the forming possibility of CNTs/Al6061 is expected to be closely associated with extrusion ratio, extrusion temperature, and CNT content.

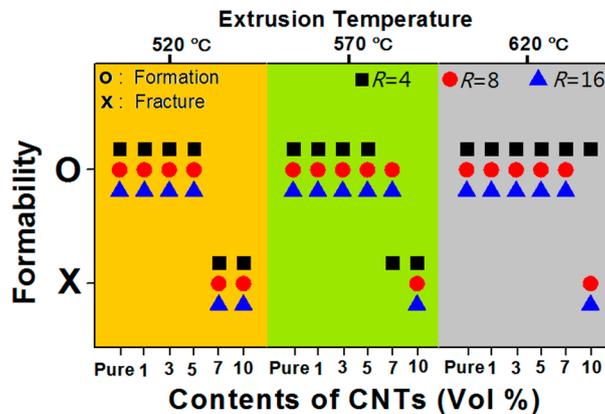


Figure 6. Formability for temperatures and ratios of extrusion (O: formation, X: fracture).

Figure 7 shows the extrusion pressure according to extrusion ratio and CNT content. It was observed that all CNTs/Al6061 composites require higher extrusion pressure than pure Al6061 alloy. As CNT content increases, extrusion pressure almost linearly increases. As the extrusion ratio increases, the extrusion pressure increases. In the case of extrusion ratio $R = 16$, the extrusion pressure exceeds 700 MPa. Owing to significant forming pressure, a damaging of the die is likely to occur, and is not appropriate for realistic application. The pressure required for extrusion forming considerably increases for composites containing greater than 5 vol % of CNTs/Al6061. It can be estimated that the increase of the CNT content and orientation requires higher forming pressure, due to the increased strength of the composite. Also, the increase of CNT content can cause a high thermal mismatch problem because of the difference in the thermal expansion coefficients between the CNTs and the

Al6061 matrix [19]. The thermal expansion mismatch could also lead to internal tensile stress. This thermal mismatch could also be a factor for increasing the extrusion pressure.

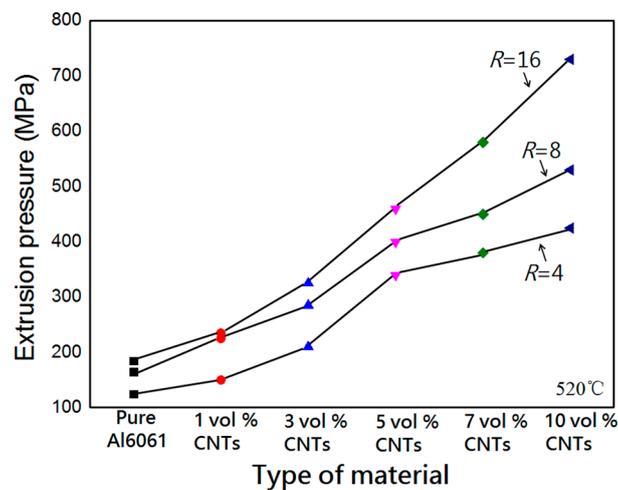


Figure 7. Extrusion pressure according to extrusion ratio and CNT content.

Figure 8 shows the variation of extrusion pressure according to extrusion temperature and CNT content. At extrusion ratio condition $R = 4$, it is observed that the pressure required for extrusion decreases with extrusion temperature. The reduction amount of extrusion pressure regarding the composites containing less than 5 vol % of CNTs/Al6061 and pure Al6061 alloy according to temperature variation is relatively smaller than that of composites containing greater than 5 vol % of CNTs/Al6061. For 10 vol % CNTs/Al6061, the forming pressure considerably decreases as extrusion temperature increases. The extrusion pressure is expected to decrease with high temperature because nears the melting temperature of the Al6061 matrix.

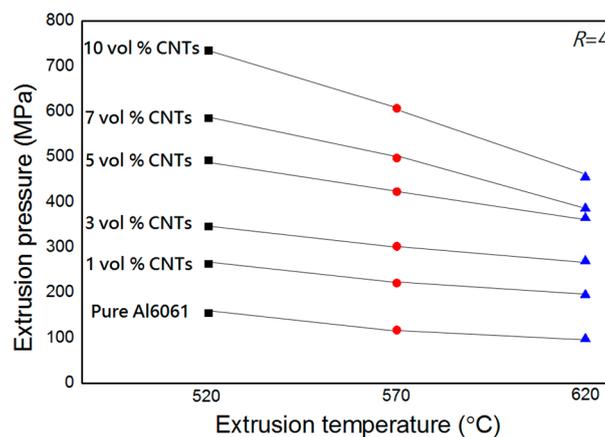


Figure 8. Extrusion pressure according to extrusion temperature and CNT content.

Figure 9 shows the Vickers hardness at three zones of an extrusion billet formed at extrusion ratio $R = 4$ and extrusion temperature 520 °C. The amount of CNT content increases as Vickers hardness increases. The highest hardness is about 230 HV of 10 vol % CNTs/Al6061 in composites. There is almost no hardness difference in the sections A, B, and C in the case of the pure Al6061 alloy. When the relative density of the preform fabricated using the powder forming process is 97%, the hardness difference for the pure Al6061 alloy is not considerable due to its equable dispersion. The CNTs/Al6061 composites with the same CNT content revealed the highest hardness in section A. The reinforcement

of CNTs generates internal pores of CNTs/Al6061 composites within the Al6061 matrix. The plastic flow by the extrusion process is analyzed as a factor causing hardness difference due to unequable CNT dispersion. To the center of Section A, normal stress was applied as hydrostatic pressure by a punch and relatively little radial stress was observed. In the edge part of section B, substantial radial stress was observed along with significant metal flow and the effects of shear stress (deformation and friction). In region C, the change in diameter is essentially complete and is expected to be primarily due to the frictional resistance of the billet sliding along the die. It is estimated that the Vicker's hardness in section B, as a compression zone, is higher than that in section A as deformation zone. Normal stress by hydrostatic pressure was applied to both section B and section C. The reason that hardness in section B is higher than that in section C is because section B was directly affected by the punch.

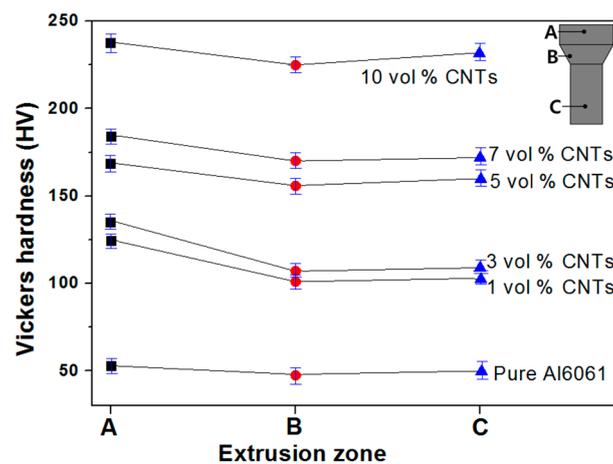


Figure 9. Vickers hardness at three extrusion zones (formed by 520 °C and $R = 4$).

The forming possibility of 10 vol % CNTs/Al6061 was investigated in further detail. Figure 10 shows the actual extrusion image in the case of $R = 4$ in extrusion ratio conditions at 520 °C of 10 vol % CNTs/Al6061 composites. This study observed the deformation behavior by dividing the extrusion part into three parts in the extrusion image. Part ① is the head part where extrusion forming is possible. Part ② is the part where surface defects are generated, and Part ③ is the part where fractures are generated.

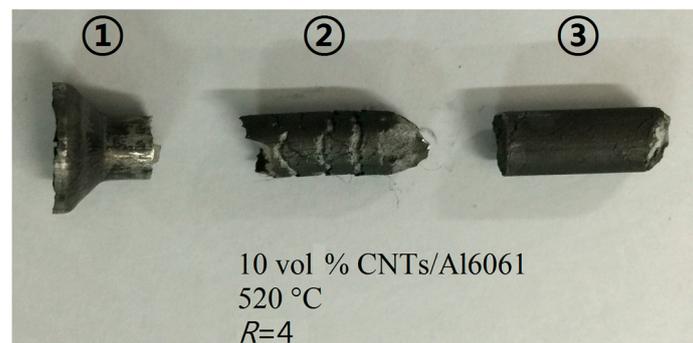


Figure 10. Physical figure of the fracture surface of extrusion part.

Figure 11 shows the variation of loads and time observed in the extrusion process (10 vol % CNTs/Al6061, $R = 4$, 520 °C). When a billet is injected into the extrusion die, pressure and time are indicated as a linear relationship, and the load subsequently increases by 2 kN/s. When the load is approximately 50 kN, the extrusion load varies for 3 min and is maintained uniformly. The extrusion

load begins to continuously increase after 3 min, and the extrusion process is preferably conducted at 70 kN.

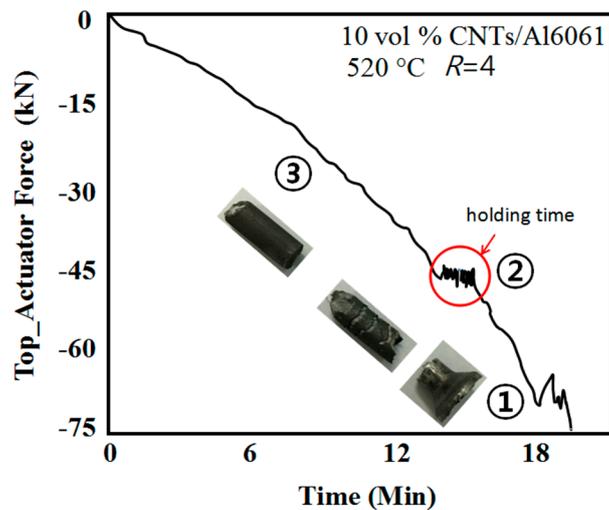


Figure 11. Top actuator force-time in the extrusion process.

The images in Figures 10 and 11 were compared for explaining the actual extrusion process. Part ③ in Figure 10 is the starting part of the extrusion process. As the plastic deformation occurs rapidly as soon as the billet passes through the final section of the extrusion die, the load increases in a linear manner. When the load reaches approximately 50 kN, no load variation is observed. In Part ②, marked with an arrow in Figure 11, an indented line defect can be observed on the extrusion part's surface. In this process, a significant extrusion force is generated, owing to a high friction in the die–billet contact part. It is estimated that the pressure required for plastic deformation is not maintained uniformly due to high friction. Moreover, a crack is generated on the extrusion part surface and, therefore, negative effects on the mechanic properties of the material are shown. It can be confirmed that the surface state is prominent, there is no surface crack, and that the forming is well-conducted in the head part of ① in Figure 10. In order to investigate the causes of forming failure for extrusion conditions 10 vol % CNTs/Al6061, 520 °C, $R = 4$, analysis of the microstructure and SEM at the fracture surface were conducted, as shown in Figure 12.

Figure 12a shows the microstructure of the extrusion billet at three positions. Section B is the edge part. Compared with the central part, large plastic deformation is generated around the edge part. The change in grain size is generated by the plastic deformation. The grains are evenly distributed around the edge part in a 45° direction according to the extrusion direction. As sections A and C are central parts, the grains are distributed well in the vertical direction. It is evident that the densification of the microstructure was conducted well by the extrusion process. Therefore, the increase of Vickers hardness occurred after the extrusion process.

Figure 12b shows the fracture surface of the 10 vol % CNTs/Al6061 composite. The CNTs disturb the deformation of the metal, thus having high strength and hardness. The part indicated by an arrow is the dimple of the fracture surface of CNTs/Al6061 composite. As there are several clusters due to the high CNT content, the fracture surface is unevenly observed from the fracture direction. The CNT clusters enhance the mechanical properties of CNTs/Al6061 composites and lead to load sharing amongst the matrix, and reinforcement due to the wettability of agglomerated CNTs in the Al matrix [20]. As the sizes of the CNT cluster are different, the internal combination of CNTs and Al6061 is not satisfactory, and uneven dispersion of powder in the powder forming stage is predicted to be a factor causing fractures.

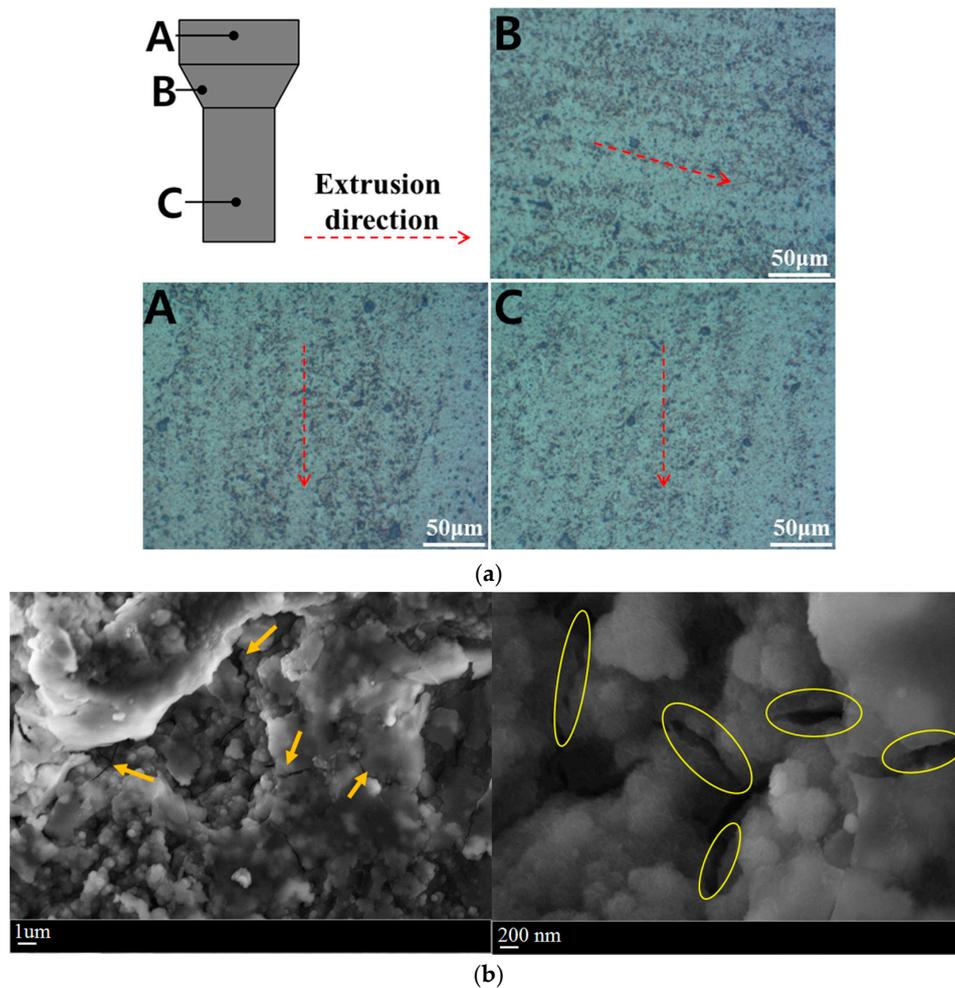


Figure 12. Microstructure of composite analysis (10 vol % CNTs/Al6061, 520 °C, $R = 4$): (a) microstructure at three positions, and (b) Scanning electron microscopy (SEM) of the dimple fracture.

The factors causing the inability to form a 10 vol % CNTs/Al6061 composite at extrusion ratio $R = 4$ at 520 °C can be analyzed as follows: First, the internal combination of Al6061 (matrix) and CNTs (reinforcement) is significantly important. Second, densification chiefly determines whether powder is evenly dispersed within the billet of CNTs/Al6061 composite fabricated using the powder forming process. Friction generated in the forming process directly affects the forming. Although friction has a positive aspect, in that necessitates high forming pressure in the plastic deformation process, it easily creates surface defects in a composite. Therefore, the optimization of the forming process by properly utilizing and controlling various parameters in the forming process is significantly important.

The densification effect after extrusion can be confirmed through the microstructure of each material. Figure 13 compares the microstructure before and after extrusion. Before extrusion, it is confirmed that several pores are included, owing to the lower relative density of CNTs/Al6061 composite compared to Al6061 material. After extrusion, the differences in the grain shape and grain size of aluminum's longitudinal section are revealed. It is predicted that there is a difference in the extrusion direction, producing orthogonal force. Owing to plastic flow, plastic deformation can be obtained [21]. The grain was observed as a thin and long-shaped material passing along the extrusion direction. When the material passes through the extrusion die and gradually exits the die, the extrusion direction of the material is limited by the die shape. Therefore, deformation of the material is not generated, and thus it has no other choice but to exit in the orthogonal direction of extrusion. In this regard, the flow of the metal is directed toward the orthogonal direction, and the grain forms a thin

and long shape. Thus, anisotropic problems of the material are likely to occur [22]. The open shape of pores before extrusion was observed according to the extrusion direction.

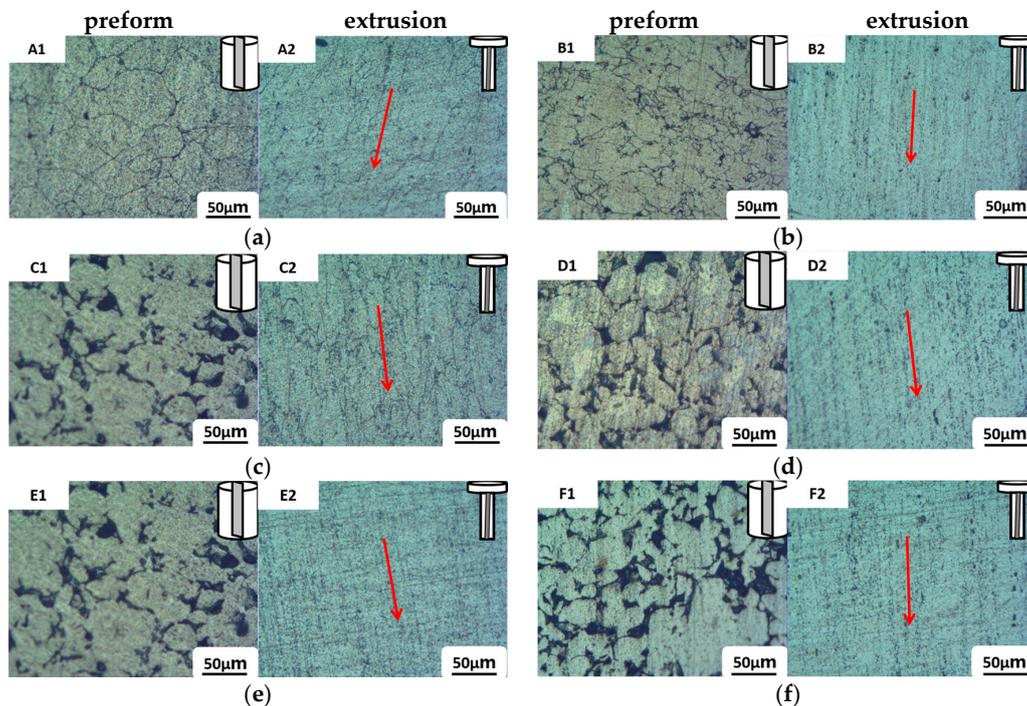


Figure 13. Microstructures after extrusion (520 °C, $R = 4$): (a) Pure Al6061; (b) 1 vol %; (c) 3 vol %; (d) 5 vol %; (e) 7 vol %, and (f) 10 vol %.

Figure 14 shows the variation in compressive stress based on the extrusion ratio. A sharp increase of compression stress in the elastic range can be confirmed, related to the densification effect during extrusion. The elongation increases until the fracture of the CNTs/Al6061 composite.

Upon comparing the CNTs/Al6061 composites and pure Al6061 alloy according to extrusion ratio, it is observed that the variation according to extrusion ratio is small in the cases of pure Al6061 alloy, 1 vol % CNTs/Al6061, and 3 vol % CNTs/Al6061 composites. When the content of CNT exceeds 5 vol %, there is a significant difference in the compression strength according to extrusion ratio. When the extrusion ratio is $R = 4$, the compression strength of 5 vol % CNTs/Al6061 and 7 vol % CNTs/Al6061 composites is almost similar. However, the compression strength of 7 vol % CNTs/Al6061 composite considerably increases when the extrusion ratio is $R = 8$. When the extrusion ratio is $R = 16$, the extrusion force of 7 vol % CNTs/Al6061 composite considerably increases. Furthermore, the compression strength of 5 vol % CNTs/Al6061 composite considerably increases, reaching 950 MPa.

Compared with all the given extrusion ratio conditions, it is observed that the variation in compression strength for 10 vol % CNTs/Al6061 composite is small. The results reveal a higher value when it is transformed to actual compression stress. This indicates that the strength and elongation of CNTs/Al6061 composites can be controlled based on the variables of formation, such as the properties of the metal used. Also, no significant differences in compression stress according to extrusion ratio were observed in terms of pure Al6061, 1 vol % CNT/Al6061, and 3 vol % CNTs/Al6061 composites. The reason is that the pressure required for their formation is small, owing to the small CNT content, and that the forming-possible space remarkably decreases, owing to the relatively higher relative density compared to the composites with high CNT content. The compression stress variation is expected to be generated by CNT distribution, CNT orientation, and the difference of the thermal expansion coefficient between the CNTs and the Al6061 matrix.

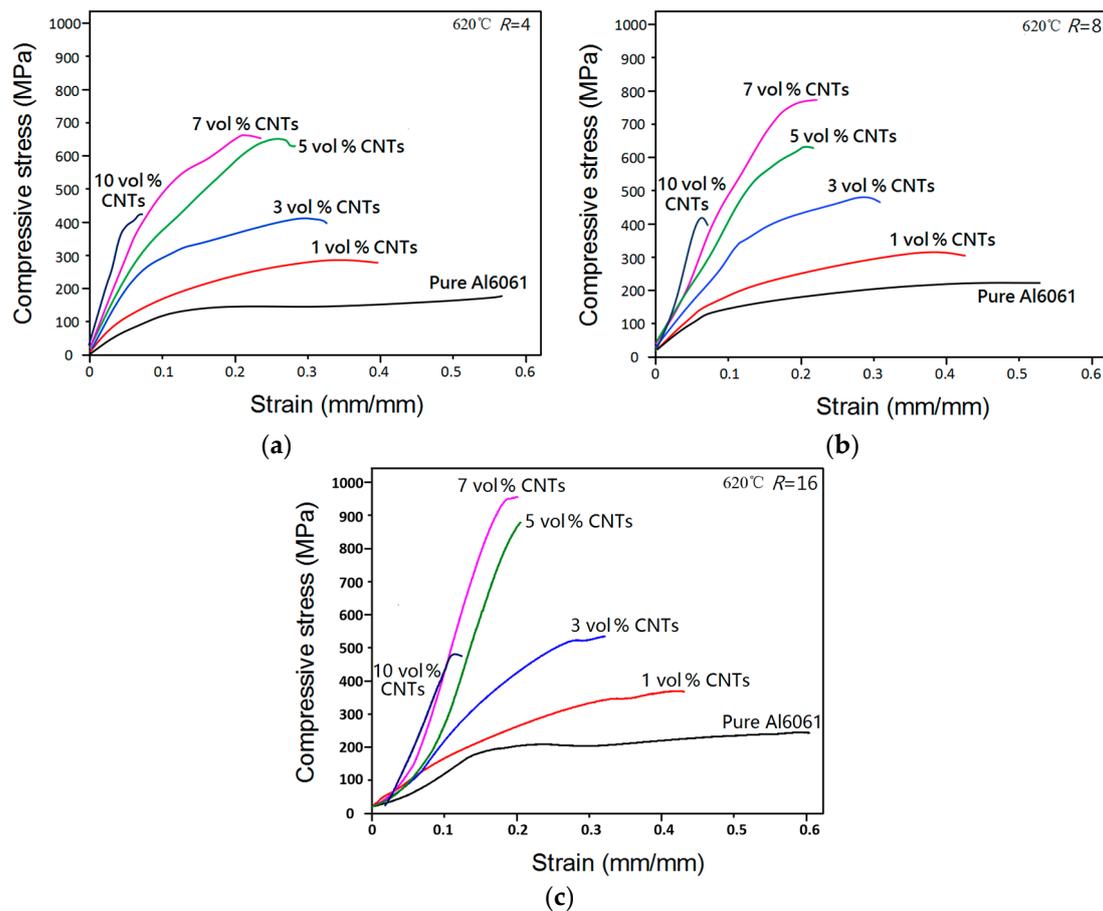


Figure 14. Compressive stress depending on extrusion ratio: (a) $R = 4$; (b) $R = 8$ and (c) $R = 16$.

4. Conclusions

- (1) The mechanical properties of composites can be further enhanced using the densification process via hot extrusion without sintering of CNTs/Al6061 preforms as a result of this study. The conclusions of this study can be presented as follows:
- (2) Using the hot extrusion process, densification is possible for CNTs/Al6061 composites with relative density lower than that of the existing Al6061 alloy. Moreover, the relative density is obtained using the hot extrusion process for preformation.
- (3) The formability increases as the extrusion temperature increases when the CNT content is low. For 620 °C, the forming of all materials except for 10 vol % CNTs/Al6061 was possible at extrusion ratios $R = 4$, $R = 8$, and $R = 16$.
- (4) As the amount of CNT content and extrusion ratio increase, extrusion pressure increases.
- (5) The amount of CNT content increases as Vickers hardness increases. The hardness of 10 vol % CNTs/Al6061 is about 230 HV and that of 1 vol % CNTs/Al6061 is about 100 HV.
- (6) The variation in compression strength for 10 vol % CNTs/Al6061 composite is small according to extrusion ratio. No significant differences of compression stress according to extrusion ratio were observed in terms of pure Al6061, 1 vol % CNT/Al6061, and 3 vol % CNTs/Al6061 composites.

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Conflicts of Interest: The authors declare no conflict of interests.

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