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Research Article

Production of Ni-GNP/AlSi12 Composite Foams and Investigation of Their Properties

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ABSTRACT: In this study, Ni-GNP/AlSi12 composite foams were produced by using powder metallurgy process using aluminum powder as matrix material, silicon (12%) for alloying, titanium hydride (1%-TiH_2) as foaming agent and graphene nanoplatelets (0%, 0.4%, 0.8% and 1.2%-GNP) as reinforcement. The powders were mixed at specified ratios, cold compressed and extruded at different ratios to produce precursor samples with diameters of 6 mm and 12 mm. Foaming experiments were carried out at 750 °C in open atmosphere by measuring the expansion rates volumetrically. After foaming, the samples were analyzed in terms of their cellular structure, relative density and cell wall hardness. The results showed that the addition of Ni-GNP decreased the expansion rates but increased the relative density and stability, and the optimum reinforcement effects were observed at 0.4% Ni-GNP concentration. Therefore, the addition of Ni-GNP provides improved stability and hardness, albeit at the expense of reduced expansion, for applications requiring structural strength.

Keywords: AlSi12 foam, Ni-GNP, Powder metallurgy, Expansion

ÖZET: Bu çalışmada, matris malzemesi olarak alüminyum tozu, alaşımlama için silikon (12%), köpürtme maddesi olarak titanyum hidrit (1%-TiH_2) ve takviye maddesi olarak grafen nanoplakacıkları (%0, %0.4, %0.8 ve %1.2-GNP) kullanılarak toz metalurjisi işlemi kullanılarak Ni-GNP/AlSi12 kompozit köpükler üretilmiştir. Tozlar belirtilen oranlarda karıştırılmış, soğukta sıkıştırılmış ve farklı oranlarda ekstrüde edilerek 6 mm ve 12 mm çaplarında öncül numuneler üretilmiştir. Köpürtme deneyleri, genleşme oranları hacimsel olarak ölçülerek açık atmosferde 750°C'de gerçekleştirilmiştir. Köpürtme sonrasında numuneler hücresel yapıları, bağıl yoğunlukları ve hücre duvarı sertlikleri açısından analiz edilmiştir. Sonuçlar, Ni-GNP ilavesinin genleşme oranlarını azalttığını ancak bağıl yoğunluğu ve kararlılığı artırdığını, optimum takviye etkilerinin %0,4 Ni-GNP konsantrasyonunda gözlemlendiğini göstermiştir. Bu nedenle, Ni-GNP ilavesi, yapısal dayanıklılık gerektiren uygulamalar için, genleşmenin azalması pahasına da olsa, gelişmiş kararlılık ve sertlik sağlamaktadır.

Anahtar Kelimeler: AlSi12 köpük, Ni-GNP, Toz metalurjisi, Genleşme

1. INTRODUCTION

Metal foams are applied in structural and functional areas thanks to their lightweight structure and unique properties. Production processes for metal foams need to be reliable, repeatable and cost-effective to ensure market competitiveness [1]. However, as the applications of aluminum foams continue to advance, higher requirements for their mechanical properties are emerging. Adding a reinforcement phase to aluminum foams is an excellent method to improve performance [2]. For example, oxide network particles in metal powder compacts help stabilize the foam structure by forming a spherical network, acting as a mechanical barrier and stabilizing separation pressure strongly [3].

On the other hand, adding TiB2 reduced the cell wall thickness in aluminum foams, preventing cell coarsening and leading to smaller pore sizes. Therefore, the composite foams showed higher yield stress and energy absorption than pure Al foams [4]. Submicron-sized MgAl2O4 particles play an important role in developing foamable aluminum alloy composites. The MgAl2O4 particles in the composite were produced through the reaction between the aluminum-magnesium alloy melt and silica particles, and a stable foam structure was obtained [5]. On the other hand, short copper-coated carbon fibers stabilize the aluminum foam by preventing cell wall rupture and reducing coalescence [6]. As can be seen from the studies, there has been a growing interest in the fabrication of aluminum matrix composite foams (AMCFs) with nanoscale reinforcements such as ceramic nanoparticles and carbon nanomaterials. In particular, Al-Si foams as matrix are widely preferred due to their superior fluidity, adequate pore structure and ideal mechanical properties [7-9]. Oversized and irregular eutectic silicon (Si) can lead to the formation of cracks. These cracks adversely affect the toughness and strength of aluminum foams. However, recent research has proven to be a highly effective solution to improve the mechanical properties of Al-Si foams, as the addition of nano-reinforcements can refine Si precipitates or pore morphology [10-14]. The intragranular and grain boundary distributions of nanoparticles in TiCNp/Al-11Si

composites by the addition of TiCN nanoparticles to molten Al-11Si alloy by ultrasonic vibrations helped to restrict the growth of α-Al and eutectic Si. The 2% volume TiCNp/Al-11Si composite showed significant increases in hardness, tensile strength and yield strength compared to the matrix alloy, while elongation improved by 108% [15]. In the study by Du et al. [16], 1% SiC nanoparticle reinforcement by volume reduced the average cell size of aluminum composite foams by 50.4%. It reduced the pore diameters to a micrometer scale.

Furthermore, the addition of nanoparticles increased the yield stress by 194.5% and energy absorption by 69.4% compared to pure aluminum foams. The research shows that nanoparticles significantly improve foam structure and mechanical properties. In CNT-reinforced aluminum alloy composite foams using in-situ chemical vapor deposition and powder metallurgy, CNTs increase the uniformity of the pores, reducing their size and thus improving the compressive behavior. This improvement is due to CNTs increasing the nucleation sites for hydrogen and limiting bubble movement during foaming [13]. Graphene nanosheets (GNSs), a member of the graphene family of materials, have become a promising reinforcement for metal matrix composites (MMCs) due to their high tensile strength and Young's modulus, significantly enhancing the toughening effect. For example, in uniaxial compression tests on micro-columns of nanolaminate graphene (RGO)-Al composites, aligning the RGO layers in the load direction or increasing the RGO concentration strengthens the material [17].

On the other hand, in graphene nanoflakes (GNFs) reinforced Al-20Si composites produced by pressure infiltration, GNFs formed a strong bond with the Al-20Si matrix without Al4C3 formation, thereby increasing the stiffness and elastic modulus. Researchers have shown that GNFs effectively strengthen Al composites [18]. Recent studies reveal the role of GNSs in strengthening, toughening and improving the structure of not only dense Al composites, but also Al composite foams. For example, in aluminum composite foams reinforced using GNSs coated with copper nanoparticles, 0.75% GNSs@Cu content increased the yield stress, plateau stress and energy absorption capacity by more than 100% compared to pure aluminum foams. This is due to the effectiveness of load transfer and dispersion reinforcement mechanisms [19]. GNFs contribute to the improvement of the pore morphology of aluminum foams and unlike other brittle foams, they can exhibit a smooth stress-strain curve. The addition of 0.10% GNFs slightly improved plateau stress, energy absorption and specific energy absorption [20].

This study provides a first attempt at evaluating the impact of Ni-coated GNPs on aluminum foams' stability and durability properties. The main challenges in producing nanocomposites are the high surface-to-volume ratio and the low wettability of ceramic particles by aluminum. Small particles tend to aggregate and lose their ability to inhibit dislocation motion. In this study, the graphene used was chemically coated with Ni and Ni-GNPs/Al-Si composites were fabricated by powder metallurgy. The effects of Ni-GNPs on expansion behavior, macro structures and the cell wall hardness of Al-Si alloy composite foams were systematically investigated. To the best of our knowledge, no previous study in the literature has explored the use of Ni-coated graphene nanoplatelets in Al-Si composite foams, highlighting the novelty of this research.

2. MATERIAL AND METHOD

In the experimental studies, the materials given in Table 1 were used as starting materials. Scanning electron microscope images of the powders used are shown in Figure 1. In addition, the chemical procedure was followed for the coating of GNP particles. The materials and procedure given in Ref. 21 were followed for this process. After the coating process, the materials shown in Table 1 were mixed in the specified ratios, cold compressed and subjected to extrusion at two different extrusion ratios (1:5 and 1:20) to produce foamable precursor samples with 6 mm and 12 mm diameters. To evaluate the foaming properties of the precursor materials produced by the hot extrusion process, the samples were cut into small pieces 10 mm in length. Subsequently, a series of foaming experiments were carried out at an oven temperature of 750°C without using a protective atmosphere (Fig. 2). The foaming experiments were observed and video recorded through a high-temperature tempered glass mounted on the oven door.

Table 1. Starting powders and their properties

Material	Function	Purity	Particle Size	Ratio (wt.%)
Aluminum powder	Matrix material	99.8%	< 44 µm	Remain
Silicon powder	Alloying element	-	< 44 µm	12
Titanium hydride (TiH_2) powder	Foaming agent	-	< 44 µm	1
Graphene nanoplatelets (GNPs)	Reinforcement element	99.9%	Thickness: 3 nm	0.4, 0.8, 1.2

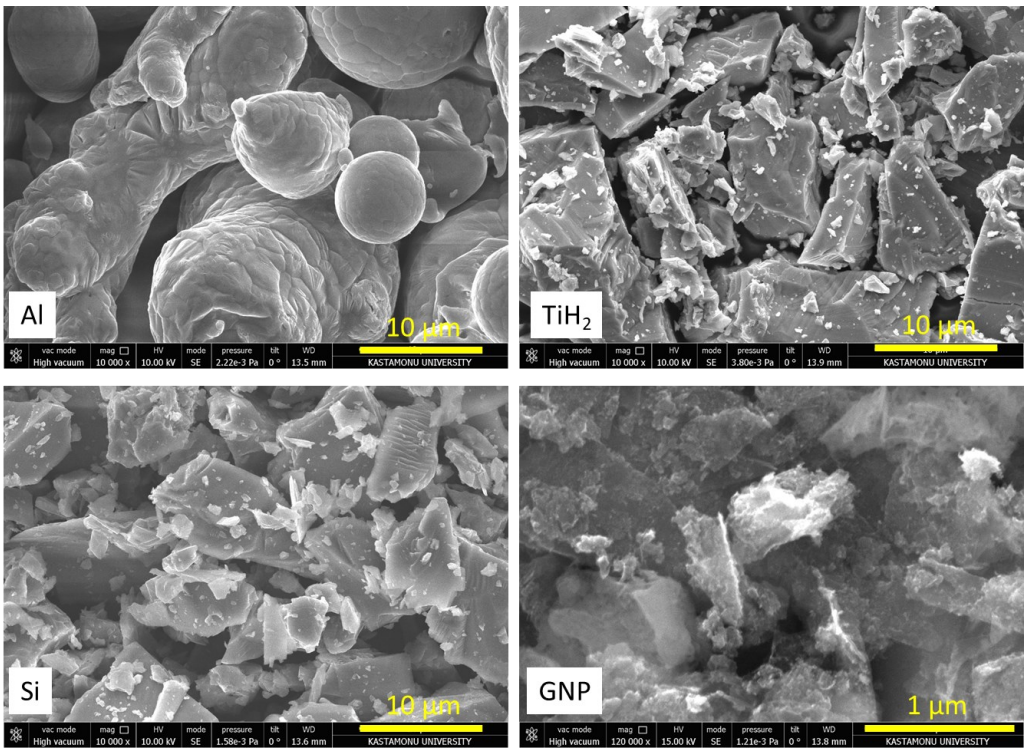

Figure 1. SEM images of the powders

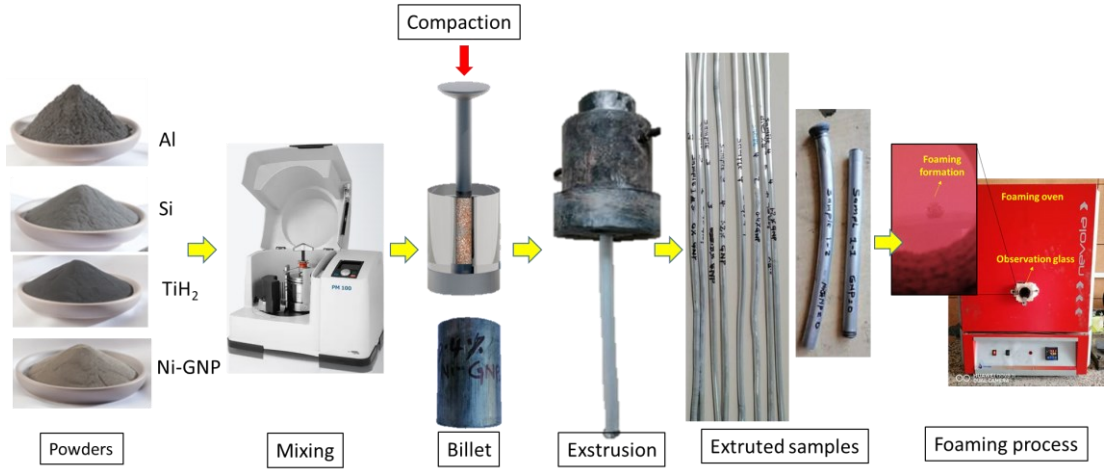

Figure 2. Schematic representation of the fabrication process of Ni-GNP/AlSi12 composite foam

After the foaming process, Ni-GNP/AlSi12 composite foams were cut in half vertically along the expansion direction, and the cut surface was polished using conventional metallography processes. Thus, it was made ready for macro examinations. The Vickers microhardness testing machine (Schimadzu (HMV-G)) was used under a 100 g load to determine the cell wall hardness of the prepared foam samples. Five measurements were made from the prepared cell wall cross-sectional surface and their averages were evaluated. Relative density (ρ^*) values were obtained as the ratio of the experimental density to the theoretical density (Equation 1).

$$\rho * = {}^{\rho_s}/_{\rho_t} \times 100\% \quad (1)$$

The expansion rates of Ni-GNP/AlSi12 precursor materials after foaming were calculated using Equation 2 below.

$$Expansion\ rate\ (\%) = \left(\left(\frac{V_{foam}}{V_{precursor}}\right) - 1\right) \times 100 \quad (1)$$

V_{foam} is the volume of the foam, and $V_{precursor}$ is the volume of the precursor material.

3. RESULTS AND DISCUSSION

3.1. Expansion Behavior

The most important feature of metallic foams is their lightweight due to their porous structure. When Ni-GNP/AlSi12 precursors obtained by powder metallurgy are subjected to foaming in the free state, the shape of the foams is irregular, which makes it challenging to evaluate the density of metal foams by the standard method. In this respect, the densities of the foams were determined using a density kit integrated into a balance with a precision of 0.0001 g. The expansion rates of the foams were determined volumetrically. Figure 3 shows the expansion ratios obtained by subjecting the foamable Ni-GNP/AlSi12 precursor materials with 6 and 12 mm diameters to free-state foaming at 750°C. As can be seen in Figure 3a, there are significant differences in the expansion behavior of the samples containing different ratios of Ni-GNP. It is noteworthy that the increase in the amount of Ni-GNP negatively affects the expansion behavior of the precursor samples.

The maximum expansion occurred in the 0 % Ni-GNP/AlSi12 precursor samples at 325%. The minimum expansion occurred in 1.2% Ni-GNP/AlSi12 precursor samples at 221%. There was a ~100% difference between both samples. Maximum expansion occurred in approximately 45 seconds in all of the samples. In Figure 3b, the expansion curves of the Ni-GNP/AlSi12 precursor materials with a diameter of 12 mm are pretty different from the precursor samples with a diameter of 6 mm. A clear relationship between specimen size and expansion behavior can be established. This situation is not only limited to the sample size but also reveals the effect of the extrusion rates applied during the preparation of the precursor samples. The extrusion ratio of 6 mm diameter specimens is 1:20, while the extrusion ratio of 12 mm diameter specimens is 1:5. In this case, the precursor specimens with a diameter of 6 mm are subjected to more plastic deformation during production and possible structural defects may occur. The fact that the hydrogen gas released by the decomposition of the foaming agent (TiH_2) during expansion is not sufficiently retained in the structure indicates that the expansion rates will be relatively low. In Ni-GNP/AlSi12 foamable precursor materials with a diameter of 12 mm, the maximum expansion was 450% in 0% Ni-GNP/AlSi12 precursor samples. The minimum expansion was 171% in 1.2% Ni-GNP/AlSi12 precursor samples. With the increase in expansion, the time to reach the maximum level also increased. Maximum expansion occurred in approximately 70 seconds in all samples. As with the 6 mm diameter precursor specimens, the increase in Ni-GNP amount negatively affected the expansion behavior of the 12 mm diameter precursor specimens.

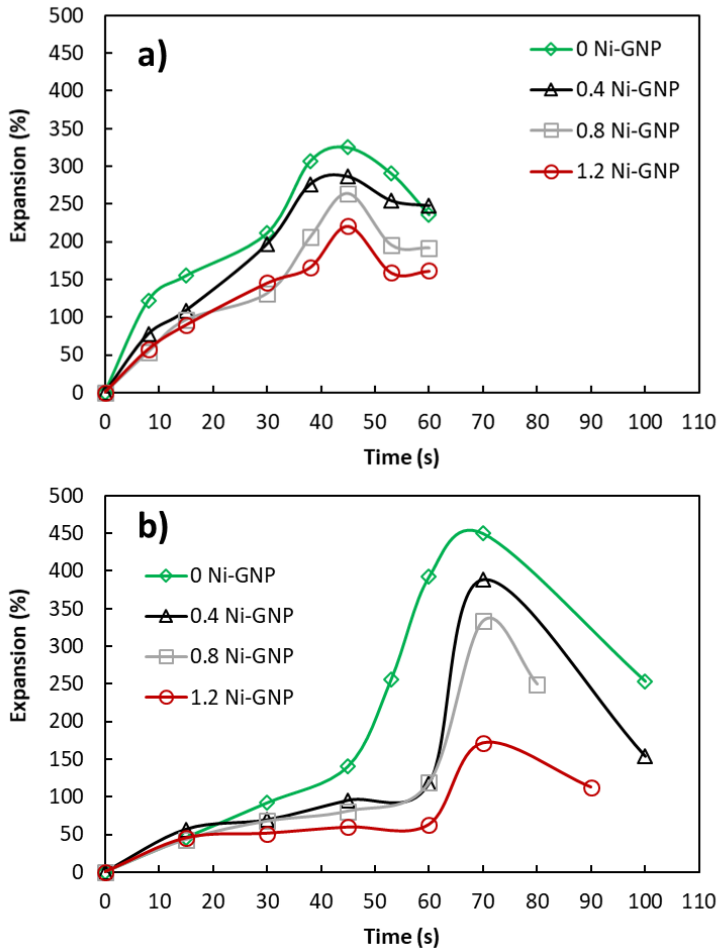

Figure 3. Expansion curves of Ni-GNP/AlSi12 precursor materials, a) 6 mm, b) 12 mm

3.2. Relative Density

Aluminum foams are materials containing a large number of pores randomly distributed within the structure. Structural properties such as pore morphology and relative density variations directly affect the mechanical properties and energy absorption capacity of aluminum foams. In fact, relative density variation is more effective than pore morphology [22, 23].

It is tough to produce products with predetermined microstructures in foams. The fact that irregularities and internal defects are always present in the structure, the cell walls are not flat, some cell walls are missing, and thickness differences occur are the factors that cause the relative densities to change [22, 24]. Table 2 shows the density variations of Ni-GNP/AlSi12 precursor samples that exhibited maximum expansion. For both 6 mm and 12 mm diameter precursor samples, the relative density values increased with the addition of Ni-GNP. However, up to 0.8% Ni-GNP addition, the relative density values of Ni-GNP/AlSi12 foams produced with 6 mm diameter precursor samples are higher than those of Ni-GNP/AlSi12 foams produced with 12 mm diameter precursor samples. The situation is different when the Ni-GNP ratio reaches 1.2%.

Table 2. Density changes of Ni-GNP/AlSi12 foams exhibiting maximum expansion

Ni-GNP ratio (%)	Experimental Density ($g.cm^{-3}$)		Relative density	
	6 mm	12 mm	6 mm	12 mm
0	0.621	0.476	0.235	0.182
0.4	0.683	0.534	0.259	0.204
0.8	0.715	0.594	0.274	0.230
1.2	0.806	0.953	0.311	0.368

3.3. *Macrostructure*

Process optimization during the foaming process is not only limited to time and temperature but also includes methods that are very difficult to control, such as taking the foaming sample out of the furnace, placing it in the cooling environment, and the effect of the environment on the solidification of the foam. Therefore, the porous structure of the final product is not controllable. The macrostructures of all Ni-GNP/AlSi12 foams produced in this study are shown in Figure 4. The structural analysis and density data for Ni-GNP/AlSi12 foams are based on the sample exhibiting maximum expansion.

The structural and physical properties of the foams, such as pore sizes (Figure 4) and expansion (Figure 3), varied significantly. The stability of AlSi12 foams can be attributed to the presence of added Ni-GNP particles. Ni-GNP particles increase the viscosity of the molten metal during expansion. For these reasons, drainage in the cell walls is reduced and the foam is stabilized [25-27]. During foaming, the walls separating the two cells rupture, resulting in the coarsening of smaller cells and the formation of large cells. The presence of large cells in foams without Ni-GNP is indicative of their poor stability. Although AlSi12 foams with the addition of Ni-GNP exhibited poorer expansion, the presence of better cell structure indicates higher stability in these foams. The stability of foams is affected by the size and distribution of particles and wetting behavior [25, 28].

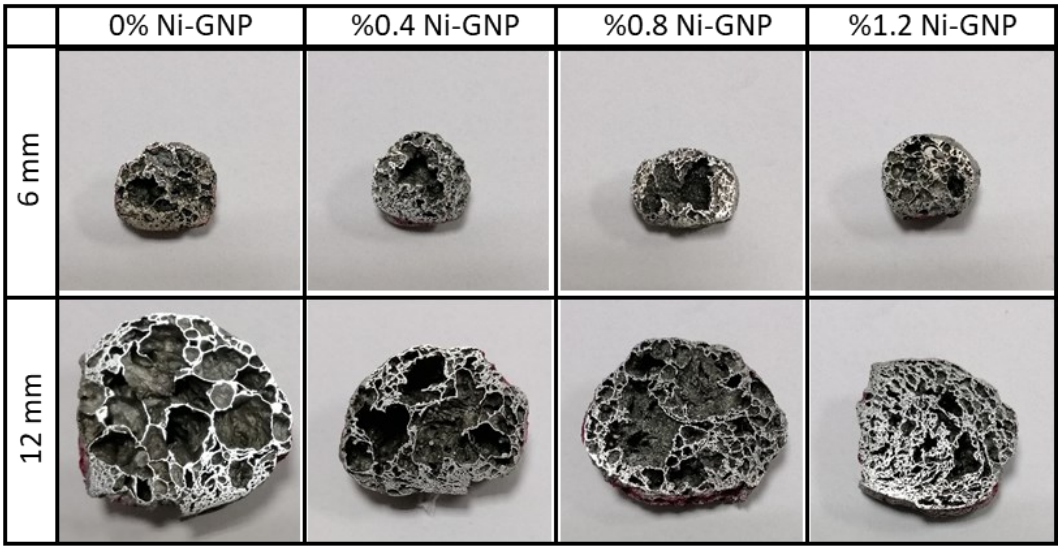

Figure 4. Macrostructures of Ni-GNP/AlSi12 foams exhibiting maximum expansion

3.4. Cell Wall Hardness

Figure 5 shows the changing trend of the microhardness of the cell walls. It is noted that Ni-GNP particles have a significant effect on the microhardness of cell walls. In general, Ni-GNP particles increased the cell wall microhardness up to 0.4%. After that, the microhardness decreased with increasing Ni-GNP content. As a result, the relationship between microhardness and Ni-GNP content is not linear.

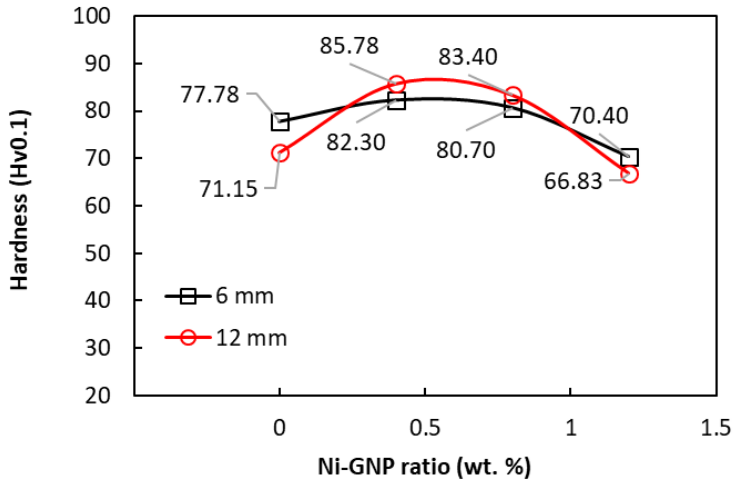

Figure 5. Cell wall hardness of Ni-GNP/AlSi12 foams

4. CONCLUSION

According to the experimental studies, the results show that the incorporation of Ni-GNP particles into AlSi12 foams significantly affects the expansion behavior, relative density, macrostructure and cell wall hardness of the obtained foams. The addition of Ni-GNP negatively affected the expansion of the foams. While the foams without Ni-GNP reached a maximum expansion ratio of 450%, the foams containing 1.2% Ni-GNP expanded only 171%. This reduction in expansion is attributed to the structural reinforcement provided by the Ni-GNP particles, which prevents the overall expansion of the material during the foaming process. The relative density of the foams increased, especially in the samples with higher Ni-GNP content. This increase in density is related to the ability of the particles to limit expansion, resulting in denser cell walls. In particular, the 12 mm diameter samples containing 1.2% Ni-GNP had the highest relative density of 0.368. The addition of Ni-GNP reduced the expansion while increasing the foam stability. Foams containing Ni-GNP exhibited more uniform pore structures, indicating improved cell wall stability. This stability is due to the increase in viscosity induced by Ni-GNP, which reduces drainage from the cell walls and maintains foam integrity. In conclusion, Ni-GNP particles appear to be useful for creating stable structures in metal foams. These findings highlight the potential engineering applications of Ni-GNP-reinforced AlSi12 foams, particularly in energy-absorbing structures such as crash absorbers or impact-resistant materials, where high stability and uniformity are essential.

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