

Mechanical and Microstructural Analysis of Nickel Foam Via Powder Metallurgy

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ABSTRACT

Materials with superior mechanical and thermal performance are important in contemporary engineering due to their lightness. Nickel foam is a promising candidate because of its high porosity, interconnected pore structure, and mechanical integrity. In this research work, nickel foam was synthesised by the powder metallurgy space holder method. Elemental nickel powder was mixed with ammonium hydrogen carbonate, which acted as a pore-forming material. The fabrication process included powder mixing, uniaxial compaction, and high-temperature sintering under an inert atmosphere. The foams that were produced had homogenous interlocked pores ranging from 11-14 μm . The results of the mechanical test indicated that an increase in sintering temperature from 660°C to 760°C enhanced strength and hardness. The crushing load and hardness of Ni78SH22 increased, respectively, from 750 N and 32.7 BHN to 850 N and 34.5 BHN, and Ni68SH32 and Ni58SH42 obeyed the same trend. The experiment showed that this processing route generated nickel foams with homogeneous pore structure and promising mechanical characteristics. The results indicate that powder metallurgy is an effective and convenient technique to produce nickel foams for use in energy systems, thermal management, and catalytic reactors.

Keywords: NH_4HCO_3 , Pore size, Powder Metallurgy, Mechanical properties, SEM.



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1. Introduction

Nickel foam is a porous metallic material with a three-dimensional interconnected structure and a high surface area, low density, and a remarkable combination of thermal, electrical, and mechanical properties. Such features render nickel foam very desirable for a variety of engineering applications that demand multifunctionality. A nickel foam made by powder metallurgy has become an efficient candidate to be applied in various applications. It may serve as electrodes in fuel cells, nickel metal hydride (NiMH) batteries, and lithium-ion batteries; catalyst support in chemical reactors; vibration damping and crash-resistant structures in the automotive and aerospace industries; gas and liquid purification filters; and thermal management elements in compact heat exchangers. Additionally, providing tunable mechanical compatibility with bone, nickel foam can serve as a biomedical implant and tissue engineering scaffold. Much has been written about the basic properties and the broad range of applications of metallic foams. Banhart [1] gave a pioneering review regarding the production and use of cellular metals, not only explaining the basic processing techniques like powder metallurgy, melt foaming, and replication, but also detailing their contribution to reduction in weight, crashworthiness, and multifunctional design. Simone and Gibson [2] highlighted the fact that porosity, pore geometry, and relative density have a strong relationship with stiffness, strength, and energy absorption of metallic foams and thus have provided a theoretical framework that is still used to direct modern foam design. Yamada et al. [3] prepared nickel foams by the space holder technique and found that pore orientation created much

anisotropy in compressive behaviour, a result that emphasises the role of microstructural control in achieving reliable performance. Qin et al. [4] have applied the principles of nickel foams to generate electromagnetic absorbers and demonstrated that, through the engineering of porosity, suitable ultrabroadband performance can be realised to support next-generation communication and stealth technologies. Rahman et al. [5] performed a study where they dealloyed Ni–Al alloys, producing nanoporous nickel foams with nanoscale pore networks of extremely high surface activity and therefore promising catalyst supports and electrochemical electrode materials. Cole and Sherman [6] addressed the application of lightweight foams as a solution to enhance the fuel economy and reduce emissions in the automotive industry, where it was observed that using metallic foams in place of conventional structural components could offer better crash energy absorption and thus improve both performance and sustainability. Rabiei et al. [7] studied composite metal foams and reported that with the combination of metals and either ceramic or polymeric reinforcements, the strength achievement and energy absorption are much higher than with pure foams; a new direction emerged in structural applications. As shown by Oh et al. [8], alloy foams of Ni and Fe were scalable by using powder metallurgy, which demonstrated industrial viability and facilitated a transition between the small-scale research in laboratories and large-scale manufacturing. Han et al. [9] assessed the incorporation of nickel foams in small-scale heat exchangers and validated their better thermal conductivity, convective effectiveness, and extended stability in service, rendering them the optimal choice in

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thermal management of electronic and aerospace apparatus. Rahman and Wen [10] examined porous Ni/NiO foams as negative electrodes in lithium-ion batteries and reported that the porous structure enabled the passage of ions and offered promising electrochemical properties, even though the foams had some limitations, like the capacity decreasing with repeated cycles. Recent research has also broadened the knowledge on foam fabrication and the properties of nickel. The authors Hafiz et al. [11] focused on nickel foams prepared using ammonium hydrogen carbonate as a space holder and also showed the effects of nickel content on microstructure and mechanical behavior. Hien et al. [12] had a review on nickel foam-based electrodes to split water and remove pollutants, with the structural engineering being the key factor to enhance functional performance. Bętkowska et al. [13] investigated the impact of porosity on the mechanical behavior of open-porous Hastelloy-X foams that would be applicable in nickel foams. Shajari et al. [14] examined the open-cell heat-treated nickel foams, in which the thermal processing influences the mechanical qualities and energy absorption. Also, the research on the influence of the sintering temperature on porous NiTi alloys [15] provides some comparative insights on the optimization of nickel foam microstructure and performance.

Despite abundant literature on the case of metallic foams, the literature has not been used to research the direct impact of the variable factors of powder metallurgy, such as nickel-space-holder ratio and sintering temperature, on the microstructure and mechanical properties of nickel foam. This limitation limits the choice of the possibility of tailoring the foam properties to some engineering demands. Therefore, one of the purposes of the present study is to address this issue by preparing nickel foams with different Ni and NH_4HCO_3 proportions (78:22, 68:32, and 58:42) and sintering temperatures (660°C and 760°C). The purpose of the work is to explore the effect of these processing parameters on the pore morphology, grain structure, and mechanical properties, as well as define the porosity-strength correlation, which may be utilized to optimize the production of nickel foam with the help of powder metallurgy.

2. Materials and Experimental Procedure

The powder metallurgy pathway produced nickel foam in this experiment using the following major raw materials: nickel powder and ammonium bicarbonate. High-purity nickel powder (average particle size 10-15 μm) and a purity of more than 99% were chosen because they are sinterable and can be easily obtained. Ammonium bicarbonate was taken as the space-holder because it decomposes at a relatively low temperature to release gases into the air and leave behind only the uncontaminated interconnected pores without contaminating the nickel meshwork. The mentioned space-holder methods are typical of powder metallurgy practice [16]. Three different compositions were prepared with varying nickel-to-space-holder ratios, namely Ni78SH22, Ni68SH32, and Ni58SH42, where SH indicates the weight percentage of the space-holder. The aim of these variations was to study, systematically, the influence of the level of porosity on the microstructural and mechanical properties of the prepared nickel foams, based on the traditional principles of preparation in powder metallurgy [17]. The fabrication process began by adding nickel powder and ammonium bicarbonate in a ball milling machine over a period of two hours to blend the powder fully without the

formation of agglomerates. A hydraulic press was used to press the hybrid powders into cylindrically shaped pellets at a pressure of 25 KN; this provided the pellets with enough green strength. Further sintering in a controlled argon environment at two temperatures, 660°C and 760°C, was performed on these green compacts to ascertain the influence of sintering temperature on densification, grain bonding, and pore structure. During sintering, the ammonium bicarbonate was lost completely to form gaseous by-products and a pore network. The nickel foams were sintered, and their pore morphology and microstructure were examined by Scanning Electron Microscopy (SEM). Compressive strength was measured in a Universal Testing Machine (UTM). An assessment of hardness was made by a Brinell hardness tester. These tests provided significant data on the relationship between the process parameters and porosity and resultant mechanical performance of the nickel foams. The procedure is illustrated in Figure 1.

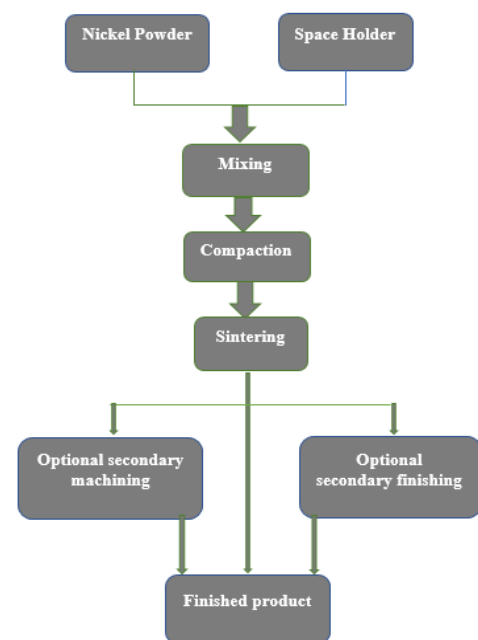


Fig. 1: Flow chart of the methodology of nickel foam production.

3. Fabrication Process

3.1 Mixing of Nickel Powder with Space Holder

When using the powder metallurgy process, the first step in the preparation of nickel foam was to combine nickel powder and a space holder. 99.48% pure nickel powder with a mean particle size of 12 μm . NH_4HCO_3 as a space holder had a melting point of 49.5°C. The 78 weight percent nickel powder and the 22 weight percent ammonium hydrogen carbonate were individually weighed on an electronic balance and then mixed. Likewise, nickel powder 68 weight percent and ammonium hydrogen carbonate 32 weight percent, and nickel powder 58 weight percent and ammonium hydrogen carbonate 42 weight percent were mixed. The respective proportions of nickel and ammonium hydrogen carbonate are shown in Table 1.

3.2 Ball milling of powder mixture

Weighing of NH_4HCO_3 powder to blend the nickel powder and ammonium hydrogen carbonate properly and uniformly together, ball milling was done. A horizontal type

ball mill was used, which consisted of a mixing chamber having a cylindrical shell. The cylindrical shell made of stainless steel was 25 cm in length and 5.5 cm in diameter. The inner surface of the cylindrical shell was made of rubber. The diameter of the stainless-steel ball of 9.5 mm and the weight of 4g were used as the grinding medium. 78g of nickel powder and 22g of ammonium hydrogen carbonate (space holder) were weighed in an electronic balance and then poured into the cylindrical shell of the ball miller. The shell was filled with 7 stainless steel balls of 9.5 mm. Then the shell was rotated at 600 rpm. The ball milling of nickel powder and ammonium hydrogen carbonate was done for 60 minutes. After ball milling, the mixture of nickel powder and space holder was obtained, where the ratio of the weight of nickel powder and space holder was 78:22. In a similar manner, the mixtures of 68:32 and 58:42 were made.

Table 1: Sample along with their respective proportion.

Sample ID	Nickel (wt %)	Ammonim Hydrogen Carbonate (wt %)	Expected porosity
Ni78SH22	78	22	Low
Ni68SH32	68	32	Medium
Ni58SH42	58	42	High

3.3 Green Compaction of Powder

Consolidation of nickel powder and space holder mixture was carried out by means of compaction. A die punch assembly was used for compaction, and pressure force was applied by the hydraulic press. The die was 38.7 mm in height and 30.2 mm in diameter, a circular die with a 9.2 mm hole through it. The plunger size was a cylindrical shape of 57.7 mm length and 9.2 mm diameter. Both the die and the plunger were made of stainless steel. For the compaction process, 3 grams of the powder mixture of nickel powder and space holder were taken, which was previously prepared by ball milling. The weight ratio of nickel powder and space holder was 78 (wt%): 22 (wt%). A stainless-steel coin was placed at the bottom of the die cavity. The mixture of a nickel powder and a space holder was poured into the die cavity. Then an upper coin was placed at the top with a plunger. Then the die and plunger were placed between the jaws of the hydraulic press. Compaction was done at 25 KN for 5 minutes, and a dense semi-finished product of cylindrical shape was obtained. In this dense product, space holders were embedded into the consolidated nickel powder. In this dense object, the weight ratio of nickel and space holder was 78:22. Similarly dense objects with weight ratios of nickel powder to space-holder of 68:32 and 58:42 were made.

3.4 Sintering

After the compaction process, the semi-finished dense object was subjected to sintering. Sintering is the process of joining metals due to atomic forces. The purpose of sintering was to achieve enough mechanical strength and also decomposition of the space holder. The sintering was carried out in an electric inert tube furnace at 660°C-760°C for 300 minutes with a holding time of 120 minutes. At this temperature, the ammonium hydrogen carbonate (space holder) decomposed and left pores in the nickel. The inert atmosphere was created by supplying argon gas into the tube chamber of the furnace, and it prevented oxygen from reacting with nickel.

3.5 Post Sintering Cooling and Handling

After sintering, controlled cooling in the furnace under a constant flow of argon was applied to the compacts. This was essential to reduce thermal stress and to avoid oxidation of the nickel matrix, which may otherwise affect the mechanical performance and surface chemistry. A low cooling rate also made sure that there was no distortion of the pore morphology or of the grain structure. After the nickel foams were sintered at room temperature, great caution was taken in picking the foams out of the furnace to prevent disturbance of the porous structure. The individual specimens were gently cleaned to remove loose surface material and then stored in airtight containers to avoid contamination of the surface before further characterization. Care in the handling of porous foams after sintering was especially critical since the mechanical integrity of porous foams is very sensitive to any microstructural defects that are introduced during processing or storage. The entire process from section 3.1 to 3.5 is shown in Figure 2.

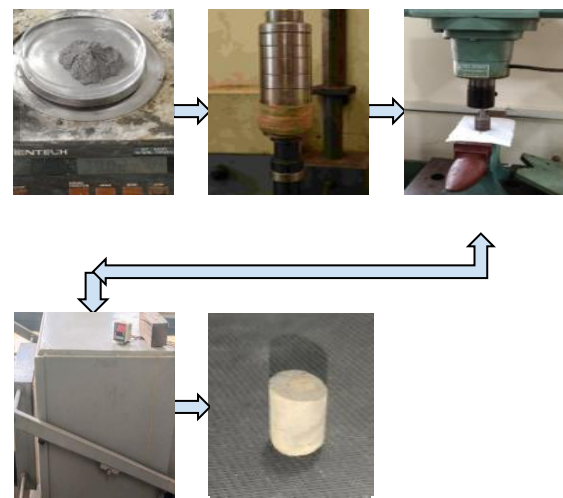


Fig. 2: Process involved in the production of nickel foam.

4. Result and Discussion

As Table 2 shows, the mechanical performance of the three alloys depends on the sintering temperature. The general trend observed in the crushing load and hardness values, with an increase in temperature from 660°C to 760°C, confirms the occurrence of an increasing trend in the values. In the case of Ni78SH22, the crushing load did rise, albeit by a small margin, from 750 N to 850 N, and the hardness also increased, albeit by a small margin, from 32.7 BHN to 34.5 BHN. This illustrates that the alloy was made mechanically stronger at an increased temperature, in both load-bearing capacity and hardness. Ni68SH32 also improved with an increase in sintering. The crushing load increased and became 750 N, and the hardness improved and became 26.2 BHN. The strength and the hardness were also improved. The same was observed with Ni58SH42, with the crushing load rising to 500 N at 760°C compared to 350 N at 660°C, and the hardness rose to 18.8 BHN from 15.8 BHN. The results refer to the fact that all alloys, even those containing less nickel, respond to a higher sintering temperature in a positive way. Similar trends have been reported in recent studies on nickel foams fabricated via powder metallurgy, where increased sintering temperature improved both crushing load and hardness due to better particle bonding and densification [11], [14].

Table 2: Crushing load, stress, and hardness values of Ni78SH22, Ni68SH32, Ni58SH42 at 660 °C and 760 °C

Alloy	Temp (°C)	Crushing Load(N)	Stress (MPa)	Hardness (BHN)
Ni78SH22	660	750	3.96	32.7
Ni78SH22	760	850	4.49	34.5
Ni68SH32	660	600	3.17	24.9
Ni68SH32	760	750	3.96	26.2
Ni58SH42	660	350	1.85	15.84
Ni58SH42	760	500	2.64	18.84

Figures 3 and 4 show how the stress and hardness vary with the sintering temperature of the three alloys. It is evident in both figures that sintering temperature raises the mechanical performance, as can be seen by the increase in stress and hardness values in all the compositions.

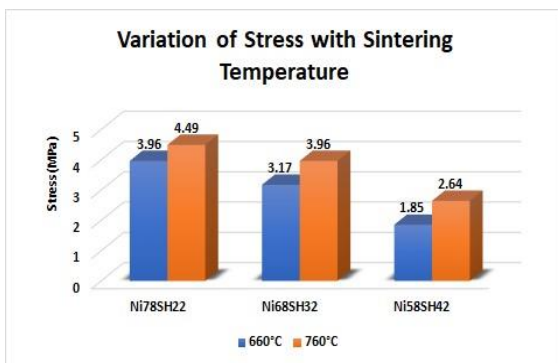


Fig 3: Variation of Stress with Sintering Temperature for different Ni-base alloys.

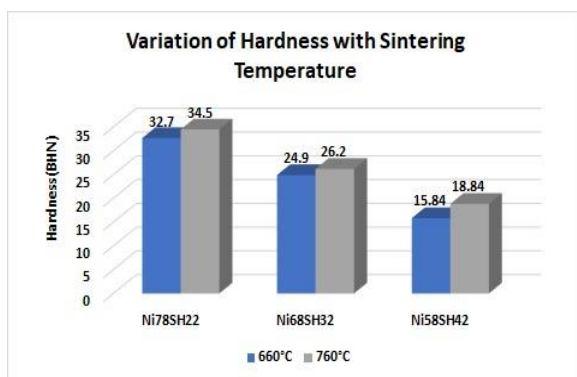
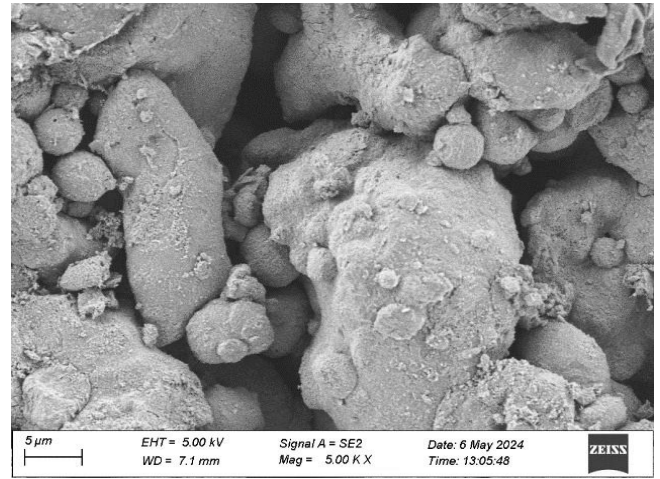
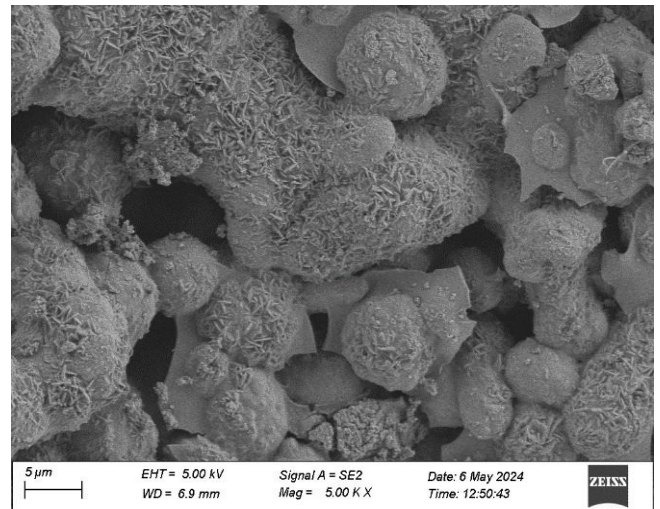


Fig 4: Variation of hardness with Sintering Temperature for a different Ni-based alloy.

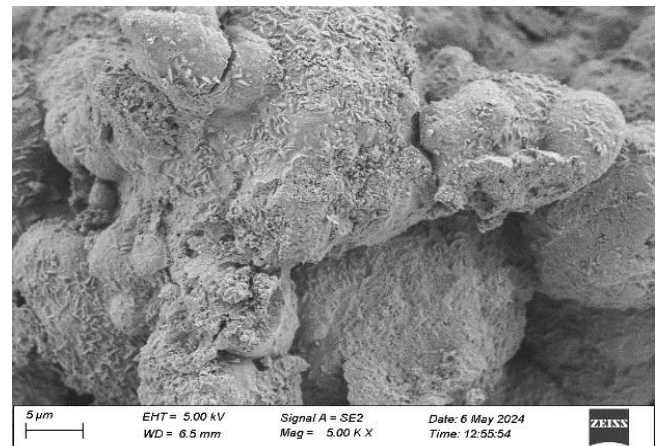
Figure 5 shows the scanning electron microscopy (SEM) images of the Ni foams produced by powder metallurgy with various proportions of nickel to space holder (NH_4HCO_3) and different sintering temperatures.



(a)



(b)



(c)

Fig.5: SEM images of Ni: NH_4HCO_3 alloys a) Ni78SH22, b) Ni68SH32, and c) Ni58SH42

It is shown in the microstructures of the samples (Figure 5a, 5b, and 5c) that processing parameters can largely affect the porosity and morphology of the nickel matrix. In all three samples, SEM analysis indicates that there is almost full densification of the nickel matrix. In the sample with a smaller space holder content (Figure 5a), the structure is arranged much more tightly, resulting in fewer and smaller pores.

The pore size and distribution tend to become more pronounced and even as the space holder content increases (Figure 5b). In the highest space holder content sample (Figure 5c), big and well-developed interconnected pores prevail in the microstructure, which shows that a greater amount of pore formation has taken place. This aligns with previous studies demonstrating that the amount of space-holder directly affects pore development and foam architecture [13], [14].

Three Ni:NH₄HCO₃ alloy compositions are presented in Figure 6. The results indicate a strong negative correlation between the space-holder content and both pore size and grain size. As shown in the graph, the sample containing the least amount of space-holder (Ni78SH22) exhibits the highest average pore size at 14.1 µm and a grain size of 13.3 µm. When the space holder content is increased to 32% in Ni68SH32, there is a slight decrease in pore size to 13.2 µm and a grain size reduction to 11.4 µm. The most significant decrease occurs in Ni58SH42, which has the highest space holder ratio of 42%. This composition shows a pore size of 11.005 µm and a well-refined grain structure with a size of just 8.068 µm. The trend indicates that an increase in space-holder content results in smaller microstructures and finer pores, leading to increased porosity. However, this increase in porosity may compromise mechanical stability, as the bonding forces between nickel particles become weaker due to reduced neck growth during the sintering process. Thus, while a higher space holder ratio can enhance functional characteristics such as permeability, it may also diminish the structural integrity of the foam. Similar inverse correlations between space-holder content and microstructural refinement have been reported in nickel foams [11], [13].

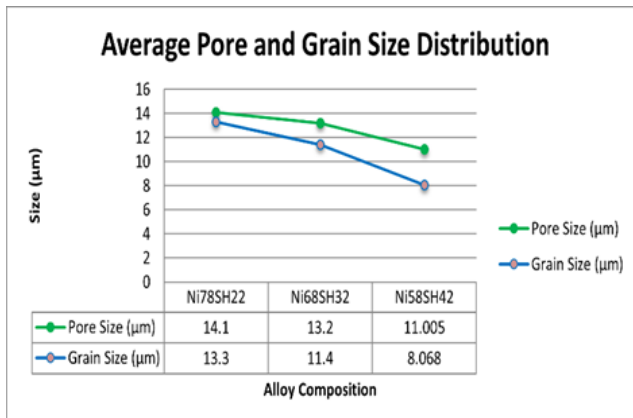


Fig. 6: Comparative pore and grain size distribution of the three alloys.

5. Conclusion

The powder metallurgy technique was used to produce nickel foam in which the space holder was ammonium hydrogen carbonate. Mechanical properties, pore morphology, and porosity were sensitive to the parameters of the process. The observation of SEMs proved the existence of a uniform porous structure incorporating pores interconnected to each other, and pore and grain size analysis was refined with the increase in the content of the space holders. It was found in mechanical testing that increased sintering temperatures resulted in increased densification and also increased crushing load and hardness values. Out of all compositions tested, Ni78SH22 was the most balanced between porosity and strength, so it can be implemented for

structural and load-bearing purposes. Ni58SH42 is not as strong, mechanically, but it is more porous, which is beneficial when it is utilized in filtration, catalysis, and electrodes. This paper proves that powder metallurgy is a viable and scalable process to produce tailored nickel foam with a specific set of properties. The development of space holder type, sintering conditions, and advanced characterization methods could help to further widen the industrial scope of nickel foam in catalysis, energy storage, and filtration systems.

6. References

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