

Characterization of Cr-Mo Alloyed Steel Foams Produced by Evaporative and Leachable Space Holder Techniques

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Abstract

Steel foams have attracted a lot of attention in both academia and industry with unique properties such as low density, high strength-to-weight ratio, operating temperature, good energy absorption, electrical conductivity, and large specific surface. The development of production methods will increase the use of steel foam. In this paper, Cr-Mo alloyed steel foams having porosities in the range of 46.8-71.3% were produced by evaporative and leachable space holder techniques in powder metallurgy. The effect on the properties of removing the carbamide used as a space holder material from the porous structure by different methods was compared. Microstructural evaluations of the pore wall, pore size, pore wall thickness, and the compressive deformation behavior of steel foam were evaluated. Steel foams produced by both routes have a rather similar macropore structure but differences in pore wall structure such as micropore ratio and pore wall thickness. The differences increase with increasing porosity content. The mechanical properties are higher in foams produced by the evaporative route as compared to the leachable route at similar porosity due to its stronger cell wall. The compressive stress and energy absorption of the leachable and evaporative process are in the range of 15-84 and 102 MPa and 1.91-6.03 and 2.98-7.83 J/mm², respectively.

1. Introduction

Metallic foam is a good candidate for structural and functional applications due to new process developments for obtaining materials with a good correlation between properties and costs [1,2]. Many metals and alloys can be produced in foam form. Among them, steel foams offer a unique combination of properties derived from their cellular structure and metallic behavior such as low density, high strength, high energy absorption, sound absorption capability, low thermal conductivity, high-temperature heat resistance, and recyclability [3]. The exclusive properties of metallic foams depend on the properties of the main alloy, porosity, the type (open, closed, and a combination of open and close), size, shape, and distribution of cells, cell wall defects, and the operational factors of the manufacturing process [4,5].

Many techniques have been developed for the production of metallic foams (direct foaming of metals,

casting, deposition, powder metallurgy routes, etc.) [6]. Among these techniques, the space holder technique in powder metallurgy is very simple and cost-effective, which can produce complex parts with easy control of pore characteristics of the foam such as porosity, pore morphology, and pore size distribution [6-9]. In this technique, the starting materials are metal powders, space holders, and binders [10]. Then, these materials go through processes such as mixing and pressing, etc. Process control is critical to achieve uniform pore structures and consistent properties, which is currently a challenge for producing metallic foams [6,11]. In this technique, the final foam structure is affected by the spacer. For this reason, it is extremely important to choose the proper space holder for the metal foam and to apply the appropriate separation process. Due to the importance of this situation, it has been included in many studies in recent years [3-21]. The space holder materials have two types, such as evaporative and leachable in a suitable solvent [6,12]. Many researchers have produced and characterized steel foams and other metallic foams through the evaporative and leachable process using space holder techniques [3,6,13]. Many

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materials are used as low-temperature evaporative space holders in the manufacturing of metal foams, such as polymer, magnesium, ammonium hydrogen carbonate, saccharose, tropical starch, and carbamide [14-18]. Furthermore, water-leachable materials such as carbamide, salt, sodium and potassium chloride and potassium bromide are generally used to produce metal foams [6,7,11,19-21]. Among the space holder materials, carbamide is very attractive for fabricating foam structures due to its low melting point, high solubility in water, and very low cost. As an additional advantage, it does not corrode the foam structure [6,11,20]. Many steel foams having different porosities and chemical compositions have been manufactured by evaporative [6,11,22] and water-leaching [6,9,19,20] techniques in powder metallurgy using irregular and spherical carbamide particles as space holders. It is observed from research in the literature that there are many advantages and disadvantages to these techniques. The water-leachable process is attractive and environmentally friendly, given that it does not lead to any dangerous by-products or emissions and does not employ any toxicity. As an additional benefit, the equipment necessary for water-leaching is very simple [6,20,22]. However, this process shows distortion in the shape and size of cells because of micro-porosities in the cell wall regions. Mechanical properties of metal foam strongly depend on cell size, aspect ratio, cell wall thickness, and micro-porosity in the cell wall surface [21,23]. Some researchers have reported that the mechanical properties of the specimens produced by the evaporative route are significantly higher than those produced by the leachable route [6,20]. However, the main problem with this evaporative process of carbamide takes a long time since a slow heating rate is required to obtain crack-free specimens, and its decomposition releases environmentally harmful gases, especially with low relative densities [23].

Many studies have emphasized that the performance of metal foams depends on the relative density, the macropore structure of the foam, and the micropores formed in the pore wall, and the pore morphology notably depends on the manufacturing process [3,4,22,24]. The aforementioned removal routes in the production process also have an intense effect on these properties of the foam structure. The use of steel foams on a large scale and successful applications of them depend on a detailed understanding of the impacts of the removal process on the properties of metal foams. The aim of this study is in this direction.

As a result of the production and characterization studies of many steel foam materials with different chemical compositions, the properties of metallic foams will be improved, and the rate of metallic foam usage will

increase. The powder grade Astaloy CrM is a water-atomized iron powder pre-alloyed with Cr and Mo exhibiting excellent hardenability. The low oxygen content gives good compressibility. Very high strength and hardness can be achieved after the sintering process. The fully pre-alloyed composition results in a homogenous microstructure. The dimensional and mechanical properties are very satisfactory. Warm compaction combined with high-temperature sintering has been outstandingly successful [25]. CrM steel foams can be used at many points as crash energy absorption, exhaust mufflers, vibration and noise control for the automotive industry, filters, heat exchangers, and high-strength wall panels for sound insulation [22]. In past years, many researchers manufactured high-density Astaloy CrM steel specimens by powder metallurgy technique and investigated the mechanical and microstructural properties [26-28]. However, no study has been found in the literature on Astaloy CrM steel foams. This paper provides the opportunity to compare the properties of Cr-Mo alloy steels based on Astaloy CrM foam and conventional materials such as Fe-Cu and Ni-Mo-Cu alloyed steel foams produced from Distaloy and Astaloy powder groups.

All these studies in the literature have shown that the "metallic foams" research area is still quite up to date and still has aspects to be researched. The adoption of steel foam relies on the manufacturing method, particularly its cost, and the resulting properties of the steel foam. In particular; the manufacturing process determines the pore morphology, properties, and performance of the metal foams. Therefore, it is important to complete the gaps in the manufacturing process. Although metal foams can be fabricated by the evaporative and leachable space holder method, it is necessary to compare the results of both routes to obtain a good foam structure. In this study, Cr and Mo based Astaloy CrM steel foams having different porosity were produced by evaporative and water-leachable space holder techniques in powder metallurgy. The structural and mechanical property variations resulting from removal from the structure of space holder particles were investigated and correlated.

2. Experimental Procedure

2.1. Materials

Cr-Mo pre-alloyed water atomized Astaloy CrM steel powder was used as parent materials, which is a registered trademark of Höganäs Company, Sweden. The chemical composition of the steel powder was 3.0 wt.% Cr, 0.5 wt.% Mo, and balance- Fe. Carbon in fine graphite form and zinc stearate were obtained from Merck Company,

Germany. 0.2 wt.% of carbon was added as fine graphite (UF4) to provide raw strength in the compression process, and it was mixed by adding 0.8 wt.% of zinc stearate as a lubricant to facilitate compression in the mold and removal after pressing. Apparent, tap and theoretical densities of the Astaloy CrM steel powders were determined to be 2.85 g/cm³, 4.55 g/cm³ and 7.78 g/cm³, respectively. The steel powders were irregularly shaped and ranged in size from 45 μm to 150 μm. The mean size distribution of the powders was 107 μm. Polyvinyl alcohol (PVA) that purchased from Merck Company (Germany) and it was used as a binder for green strength. Spherical carbamide particles were purchased from Merck Company (Germany) and were used as space holders for the ease of water-leaching or thermally. The particles had a density of 1.34 g/cm³, melting temperature of 133 °C, and solubility in water at 20 °C of more than 1000 g/L. Spherical carbamide particles were sieved to obtain a fraction of +710 -1000 μm. Figure 1 shows the typical morphologies of the irregular-shaped Astaloy CrM steel powder and the spherical-shaped carbamide particles.

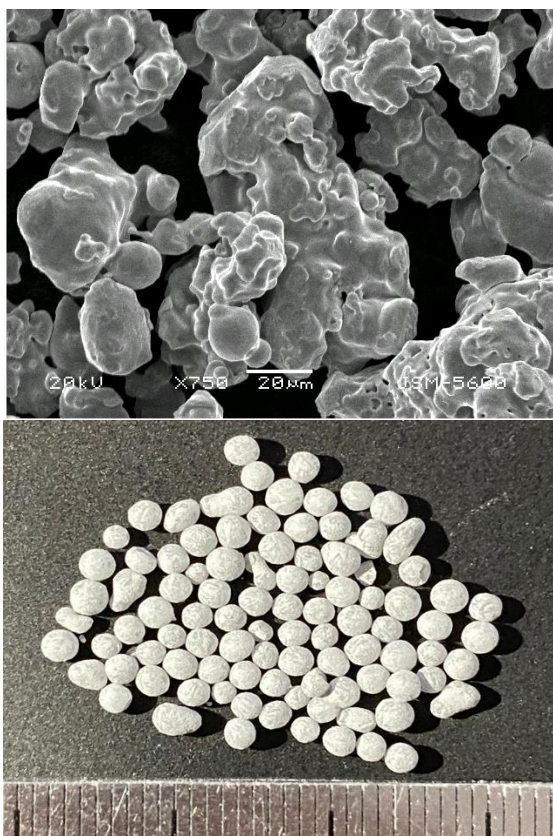


Figure 1. (a) SEM image of Astaloy CrM steel powder and (b) Photograph of carbamide particles.

2.2. Manufacturing of Cr-Mo Steel Foams

Cr-Mo alloyed steel foams were produced by

evaporative and leachable space holder techniques in powder metallurgy. PVA solution (1 wt.% PVA and 99 wt.% distilled water) was used as a binder for the green strength of the foam specimens. Initially, the steel powders were mixed by adding a 2% by weight PVA solution. Next, the PVA added steel powders mixture and carbamide particles were mixed in different volume percentages of 50, 60, 70, and 80 wt% in order to obtain specimens with different porosities. The mixture was performed in a Turbula type mixer for 60 min. The photograph of carbamide particles after coating with the steel powders is shown in Figure 2(a). A homogeneous coating of carbamide particles with the steel powders was obtained. The coated carbamide particles were then compressed with a hydraulic press machine at 200 MPa to a diameter of 10 mm and a height of about 15 mm in a cylindrical stainless steel mold. The applied compaction pressure was chosen to be 200 MPa because the green samples had lower strength at lower applied stress and the carbamide particles were fractured at higher applied pressure. The green specimens after compacting are shown in Figure 2 (b).

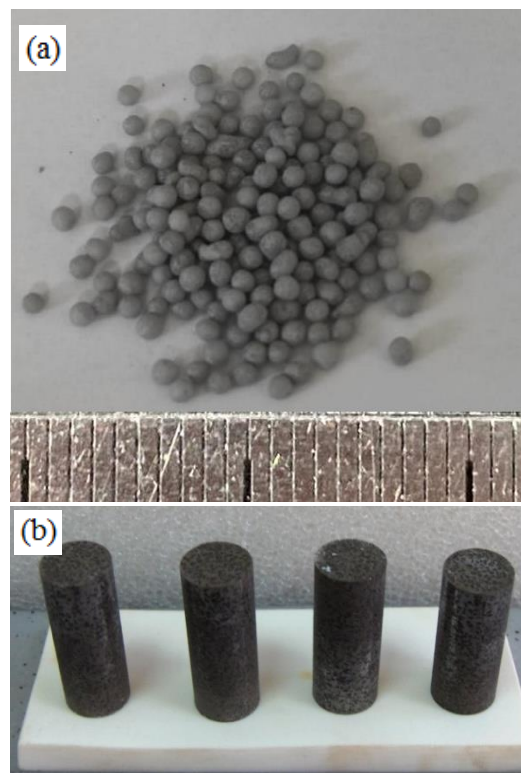


Figure 2. (a) Photograph of coated carbamide particles and (b) green specimens

After compaction to obtain a porous structure; the removal of carbamide particles was performed in two different techniques as leachable and evaporative routes. The main stages for manufacturing the Cr-Mo alloyed steel foams are schematically shown in Figure 3.

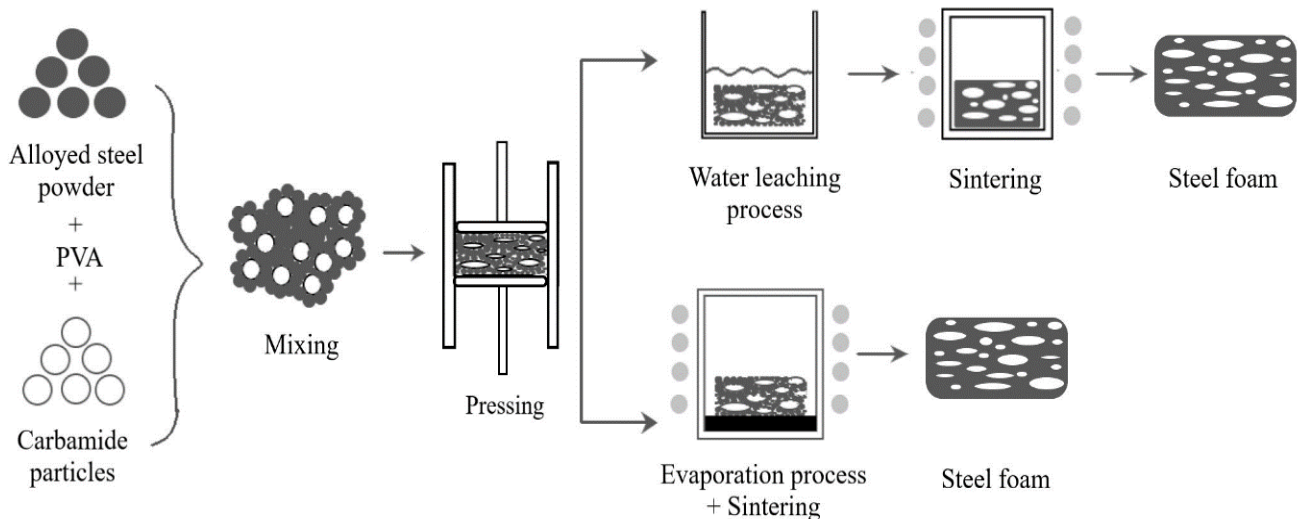


Figure 3. Schematic representation of the steel foam production process produced by leachable and evaporation techniques.

In the leachable route, the green specimens were immersed in distilled water at room temperature and held for times ranging from 30 to 120 min to leach the carbamide. The leached specimens were then rinsed with ethanol and dried in the oven at 50 °C for 2 h. More than 90% of the carbamide could be removed for theoretical porosities of 70% and 80%. About 15% to 20% of carbamide remained in the green specimens for theoretical porosities of 50% and 60%.

In the evaporative route, the green specimens were heat-treated from room temperature at 200 °C for 10 h and kept at this temperature for about 10 hours for thermal debinding of the vast majority of carbamide particles. The green porous structure was achieved by performing water-leaching and thermal decomposition of carbamide.

Both routes of techniques of removing the space holder were then heated at 450 °C for 60 min at a heating rate of 5 °C/min for further removal of PVA and remaining carbamide. Finally; all the green specimens were sintered in a laboratory tube furnace at 1150 °C for 60 min at a heating rate of 10 °C/min in an atmosphere of 50% nitrogen/50% hydrogen, with methane addition.

2.3. Characterization of Sintered Cr-Mo Foams

The density and porosity content of steel foam specimens were determined employing Archimedes' principle, using Sartorius precision balance equipped with a density-determination kit. The ratios of open and closed porosity were determined by weight measurements before and after the samples were dipped in boiling paraffin at 150 °C. The pore morphology of the foam specimens was

examined using Scanning Electron Microscopy (SEM). The size, shape, and size distribution of the pores were determined using commercial image analysis software (Clemex Vision PE). The micro porosities of the cell wall region in the steel foam samples were calculated by the Image-J software. To study the mechanical properties of the fabricated steel foams, compressive tests were performed at 0.5 mm/min cross speed at room temperature using the Zwick-Roell Z050 material testing machine.

3. Results and Discussion

3.1. The removal process of carbamide particles

The removal of carbamide can be considered as the most important part of the production step because if any carbamide remains at this stage, these carbamide residues can react with steel powder or alloying elements at sintering temperatures. As a result, the mechanical properties of the final product may deteriorate. There are many studies supporting this in the literature [6,7,20,21,29].

The increase in the temperature of the water in the leaching process caused an increase in the carbamide removal rate and caused cracking and fragmentation of the sample. Therefore, the leaching process was carried out at room temperature. Also, the leaching was done in a stagnant environment when the turbulence of the water caused the outer surfaces of the specimens to be destroyed. In the leachable route, the green specimens were immersed in distilled water at 25 °C to leach the carbamide. More than 90% of the carbamide was removed in specimens with

70% and 80% porosity by volume. About 10% to 20% carbamide remained in the green specimens with %50 and 60% porosity by volume.

In the evaporative route, various experiments were carried out considering the holding temperature, heating rate, and holding time in order to remove the carbamide from the porous structure. Previous studies in the literature have been taken into account in the removal of carbamide [6,7,20, 21,29,30]. Firstly, the samples that were heated for 2 hours and kept in a furnace previously heated up to 200°C for 2 hours were observed. However, it was observed that the gas pressure created by the sudden decomposition of the carbamide caused the sample to rupture. It was observed that the carbamide melted rapidly due to the rapid heating of the sample. The removal of powder with the resulting carbamide melt caused the specimens to deform as seen in Figure 4.

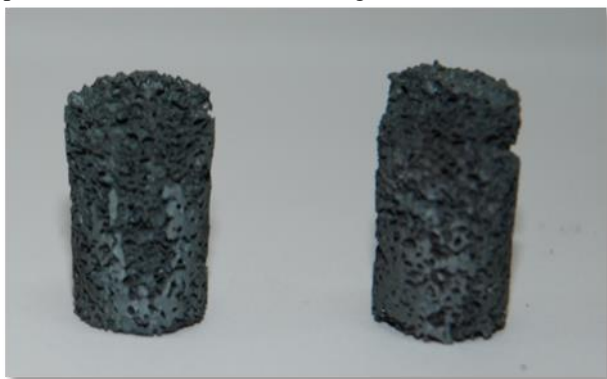


Figure 4. The deformed specimens by melting of carbamide

The melting temperature of carbamide is ~135°C but higher temperatures can be reached without melting because it needs time for the completion of melting and during that time some decomposition reactions start. Around 140°C both urea decomposition and biuret formation start, so at 150°C the carbamide removal could be possible without melting it [31]. It was observed that at 150°C after a certain period removal proceeds very slowly. Experiments showed that when the sample was heated up to 150°C and kept at this temperature observable removal started not earlier than 2 hours, and after a time period of 20 hours, only 50% of the area in the sample could be removed. Also, biuret formation cannot be prevented at this temperature, and as little dots and white particles form on the samples as shown in Figure 5. These were considered as a reformation of solid carbamide or formation of biuret from carbamide vapor. As a result, since only 50% of the carbamide could be removed after 20 hours of keeping at 150 °C this process was eliminated. In the case of 150 °C the process was slow, took 20 hours for optimum removal so 150°C was left out of consideration.

As a result; according to the experiments, the optimum pre-removal was determined as heating the sample from room temperature to ~200 °C for 10 hours and keeping it at this temperature for about 10 hours. Removal of the very small amount of remaining carbamide was removed by thermal pyrolysis during the sintering process. The selection of an appropriate thermal debinding of carbamide ensures that the carbamide particles can be removed from the green compacted specimens without breaking them [29,32].



Figure 5. Image of the sample as a result of biuret formation from carbamide vapor

3.2. The Pore Morphology and Porosity

Cr-Mo steel foams with porosities ranging between 46.3% and 71.3% were produced with both techniques. The SEM images at a different magnification of the surfaces of the foams containing about 70% porosity produced by the leachable and evaporative routes are shown in Figures 6 (a) and (b), respectively. As can be seen from the figures; two types of pores are observed in the steel foam. One type (macropore) is related to the resolved carbamide particles; the other type (micropore) is formed between the agglomerated iron particles during the sintering process. Thus, the total porosity consists of the amounts of macro and micro porosity in the pore wall. The formation of porosity at the micro and macro scales was investigated by many researchers for metallic foams [6,13,22,23,30,32]. It is observed that the specimens have a relatively uniform pore distribution. Mostly, pores are open and interconnected to each other with the pore wall. Pore walls separating each pore from its neighbors can be clearly visible. The morphology of the pores in the final products was similar to that of the carbamide particles for all the specimens. Steel foams produced by the evaporative route clearly show that the pore is interconnected and dense without any distortions in the pore wall regions.

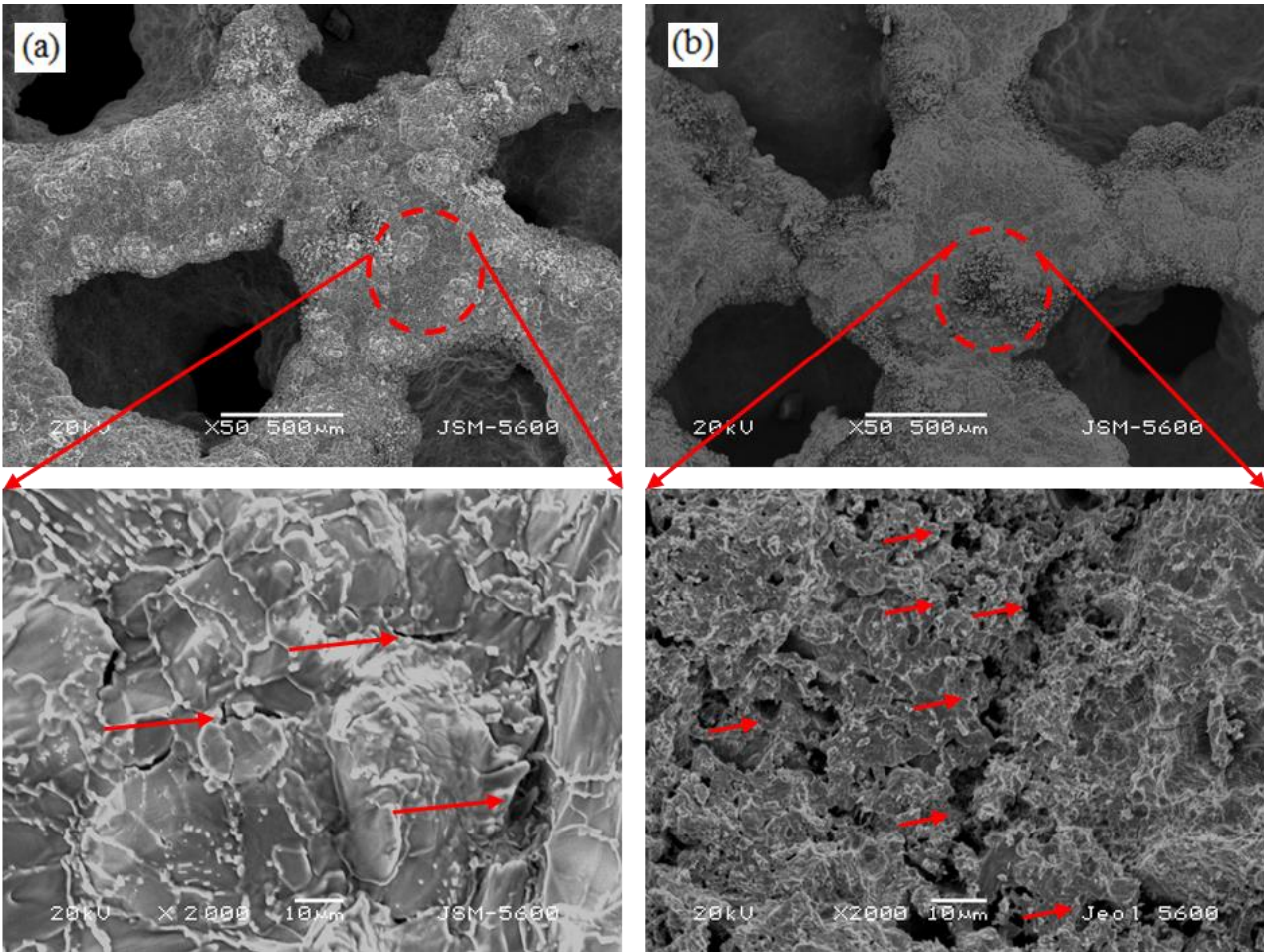


Figure 6. SEM images of 70% porosity steel foams: (a) leachable process, (b) evaporative process.

The lesser microporosity content in the pore wall regions are due to excellent localized fusion during sintering. But in the case of steel foams made by the leachable route, the highly porous specimens are distorted, and the pore wall is broken because carbamide particles are highly soluble in water. Also, numerous micropores have formed in the cell walls. These defects included cracks, holes, local porosity, and inhomogeneous composition. It has been theorized that the major occurrence of micron-level defects results from the sintering fabrication process. The interconnectivity of the foams also increases with porosity. This situation is also supported by studies dealing with similar issues in the literature [6,7,21,29,32].

Table 1 presents the density, the total porosity, the amount of open and closed porosity, the average value of microporosities, pore size, and pore wall thickness of the steel foams produced by different carbamide removal processes. In the specimens produced by both processes; it was observed that when the amount of carbamide particles used increased, the total porosity increased, and the sintered density decreased. It was determined that when the total porosity level increased in the sintered samples, the open porosity of the samples increased. The probability

of connection of the pores in sintered samples is expected to increase at high porosity levels. The pore wall thickness decreases with increasing carbamide content simultaneously due to increased pore size. The probability that the pores in the steel foam samples produced by the leachable process are connected will increase at high porosity levels. It was also found that the cell size and micro porosities in the pore wall surface are higher in steel foams produced by the leachable process compared to the evaporative process. It may be due to the higher percentage of shrinkage and a lesser micro porosity content in the pore wall surface in the case of the evaporative process. Also, this may be possible due to the less densification that occurred during the sintering process in the case of the leaching. In some studies, on the metallic foams, the macropore obtained as a result of spacer and micropore in the pore wall produced by leachable route formation has been explained similarly [6,12,21,30,32]. Jain et al. [6] reported the strong cell walls and lesser microporosities in the cell wall regions for the evaporative as compared to the leachable route. The increase in the ratio between values with the increase of the pore ratio; is very likely due to the difference in the cleanliness of the

specimens between the two methods.

Table 2 shows the mechanical properties such as compressive stress, plateau stress, and energy absorption of the steel foams produced by different carbamide removal processes. Compression test results showed that the compressive strength decreased with increasing porosity of the foam samples having different porosities. As a result of the increasing porosity, the interconnections of macropores in foams also increase, so the structure tends to be weaker under compressive load. Also, the wall thickness of the pores decreases as a result of the increased amount of total porosity, which weakens the mechanical behavior of the steel foam samples. The mechanical properties are higher in foams produced by the evaporative route as compared to the leachable route in all porosity conditions. This may be due to the larger amount of microporosity in the pore wall region caused by the dissolution of carbamide in water, and lesser particles to particle bonding in the case of leaching. The removal of the carbamide process significantly affected the plateau stress value in all porosity content. The plateau stress of steel foams produced by the

evaporative route is higher. This is probably a consequence of the less microporosity of the pore walls. The high content of micro porosities in the pore wall region makes the structure fragile and mostly distorted. When the porosity is increased, there are significant differences between the two removal methods. Some research results show that the porous steel specimens produced by the decomposition route were generally much stronger and stiffer than those produced by the dissolution route. They explained the main reason for this as the decomposition route uses a higher sintering temperature, resulting in better bonding between the steel particles [6,18,22,31,33]. Lu and Zhao [19] reported that the decomposition removal process is the best option in the terms of maximizing the stiffness and strength of the porous steel. The energy absorption depends on the porosity of the foam and increases with decreasing the porosity of the steel foam [3,6,34]. It was found that the energy absorption values are higher in the evaporative route as compared to the leachable route, and also the value of energy absorption is decreasing with an increase in porosity.

Table 1. The density, total, open and closed porosities, micro porosities in pore wall region, pore size, pore wall thickness of the steel foams.

Carbamide removal process	Carbamide fraction (vol.%)	Density (g cm ⁻³)	Total porosity (%)	Open porosity (%)	Closed porosity (%)	Micropore ratio in pore wall (%)	Pore size (µm)	Pore wall thickness (µm)
Leachable	50	4.12 ±0.12	47.2 ±1.5	32.7	14.5	5.46±0.41	975.5±158	651±45
	60	3.57 ±0.06	54.2 ±1.4	42.2	12.0	6.87±0.32	992.4±147	558±34
	70	2.97 ±0.07	62,0 ±1.6	50.2	11.8	9.48±0.38	1026.7±169	451±25
	80	2.33 ±0.05	70.2 ±1.2	62.9	7.3	11.75±0.32	1158.4±187	325±19
Evaporative	50	4.19 ±0.09	46.3 ±1.3	32.9	13.4	2.16±0.32	898.3±102	611±38
	60	3.61 ±0.08	53.7 ±1.4	43.7	10.0	2.38±0.39	976.2±124	422±35
	70	2.84 ±0.06	63.6 ±1.5	52.9	10.7	3.46±0.31	988.4±132	358±28
	80	2.24 ±0.08	71.3 ±1.7	65.7	5.6	4.16±0.30	1052.2±144	328±21

Table 2. The compressive stress, plateau stress and energy absorption of the steel foams.

Carbamide removal process	Total porosity (%)	Compressive stress (MPa)	Average plateau stress (MPa)	Energy absorption Joule/mm ²
Leachable	47.2	84±1.75	68±0.98	6.03±0.08
	54.2	68±1.82	45±2.82	4.88±0.06
	62,0	36±1.68	24±3.17	3.29±0.07
	70.2	15±2.71	9±1.84	1.91±0.05
Evaporative	46.3	102±2.71	86±1.12	7.83±0.07
	53.7	82±1.83	61±2.58	6.16±0.04
	63.6	47±1.68	38±3.81	4.28±0.06
	71.3	24±2.38	19±2.11	2.98±0.03

4. Conclusions

Cr-Mo-based steel foams having different porosity were produced by evaporative and water-leachable space

holder techniques in powder metallurgy. The effects of the removal process of carbamide on macro and micro porosity structure, microstructure, and mechanical properties were investigated and evaluated. The following

conclusions have been reached:

- In the space holder-sintering technique, the crucial step is the removal of the carbamide from the green compacts such as leachable and evaporative processes.
- Steel foams produced by both routes have a rather similar macropore structure but differences in pore wall structure such as micropore ratio and pore wall thickness.
- The presence of microporosity in the pore wall regions as a result of the carbamide removal process has a significant impact on the mechanical characteristics of the steel foams.
- The strong cell walls and lesser microporosities in the cell wall regions for the evaporative process as compared to the leachable process. Therefore; steel foam produced by the evaporative route was generally stronger and stiffer than those produced by the leachable route. The differences increased with increasing porosity content.
- As a result, if both methods are compared; the leachable process is more economical, takes a shorter time, and is an environmentally friendly method. However, the mechanical properties of the product are worse. In the evaporation process, the removal of carbamide occurs at the sintering stage of production, so it is not economical because it requires long-term work at high temperatures. In addition, the gases formed during the removal of carbamide are also harmful to the environment. But in terms of the final product, the mechanical properties result in better.

Declaration of Ethical Standards

The author of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Conflict of Interest

The author declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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