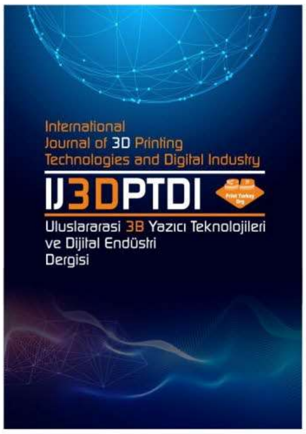

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PRODUCTION OF Ni-HARD ALLOY POWDERS BY GAS ATOMIZATION

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ABSTRACT

Ni-Hard cast iron materials are frequently used in equipment where high wear resistance is required in industrial applications. Generally, Ni-Hard is produced by conventional casting techniques. In this study, it was aimed to produce Ni-Hard powders to produce Ni-Hard cast iron materials with additive manufacturing techniques. To produce spherical and fine raw materials for additive manufacturing techniques, the gas atomization technique with a close-coupled nozzle system was preferred for the production of Ni-Hard powders. Ni-Hard alloy was melted in a high-frequency 10kW induction furnace under a protective atmosphere integrated into the gas atomization system. During the atomization process, 150 °C superheating was applied to prevent the liquid metal from freezing and clogging the melt delivery tube. In terms of the continuity of the atomization process, the negative pressure (aspiration pressure) values formed at the end of the melt delivery tube at different gas pressures were measured. An aspiration pressure of -50 mbar was obtained under an atomization pressure of 35 bar. Particle size distributions, hall flow behaviour and angle of repose properties of the produced powders were determined. Finally, the characterization of the powders was carried out by scanning electron microscopy. It was determined that the powders obtained as a result of atomization exhibited a spherical morphology and a narrow size range. The Hall flow rate test result of Ni-Hard powders was measured as 22 seconds for 50 g.

Keywords: Gas Atomization, Additive Manufacturing, Ni-Hard, Powder Characterization.

1. INTRODUCTION

Cast irons are the alloy group with the highest production capacity in the parts produced with the casting technique, which is one of the traditional production methods. Cast irons are generally Iron-Carbon-Silicon alloys with high hardness and high wear resistance. It has been classified in various ranges and properties according to its alloying elements in cast irons. The existing wear resistance and mechanical properties of high alloy white cast irons can be improved by the addition of carbide-forming elements (Nb, Mo, W, Cr, V, Ti) and a suitable heat treatment process [1]. High-alloyed white cast irons are widely used in the mining, cement, machinery and construction industries, as they have high wear resistance. Ni-Cr alloy white cast irons are called Ni-Hard alloys. High chromium white cast irons can form $M_{23}C_6$, M_7C_3, M_6C and M_3C type carbides depending on their chemical composition [2], [3]. Ni-Hard alloys are also used as the final piece after sintering. In addition, in the industry, coating with Ni-Hard is made to extend the performance and life of cutting and drilling tools. In the past, traditional coating methods such as nitriding, carburizing and electroplating were preferred in the industry. However, with the developing technology, thin film PVD and CVD coating techniques and thick coating thermal spray, laser cladding techniques appear. It has been determined that the corrosion resistance of the parts has improved in the coatings made with various alloying elements added to the Ni-Hard alloys [4]. Ni-Hard white cast irons are called Ni-Hard 1, Ni-Hard 2 and Ni-Hard 4 cast irons. Considering this classification, Ni-Hard 4 alloy has higher wear resistance and toughness strength than Ni-Hard 1 and Ni-Hard 2 alloys. For this reason, Ni-Hard 4 alloys are frequently used, especially in places where high wear resistance is required [5]. Although casting is the widely used production technique of high alloy cast irons, powder metallurgy is also a very advantageous method in producing this alloy. Despite the traditional production methods of powder metallurgy, no defects negatively affect mechanical properties such as gas voids and splitting [6]. Complex parts that casting methods cannot produce can be produced by powder metallurgical methods such as hot press (HP), hot isostatic press (HIP), laser cladding, flame spray process and additive manufacturing (AM). Especially in recent years, the need for raw materials for additive manufacturing technologies, the development of coating technologies and the use of powder as raw material have allowed atomization methods to be preferred more [7]. The powders to be used are expected to have high fluidity and different particle size distributions required according to the method. In this respect, although there are many different powder production techniques (mechanical, chemical and atomization techniques), especially gas atomization techniques are ahead of other powder production techniques. The gas atomization technique can be expressed as a conventional powder production technique for metal powder production. The fact that many different parameters take place in the gas atomization production processes, which is based on the principle of molten liquid metal flowing through a nozzle and breaking it down with different gases (air, nitrogen, argon and helium), allows the economical production of powders needed by various sectors [8-9]. In this study, Ni-Hard alloy powders were produced by gas atomization technique. The powders produced were characterized in detail.

2. MATERIAL AND METHOD

The chemical compositions of Ni-Hard (white cast) alloys, which are frequently preferred in industrial applications, are given in Table 1. Ni-Hard 4 alloy was atomized with the gas atomization system in Kocaeli University Metallurgical and Materials Engineering Department. Gas atomization system consists of a nozzle, induction melting device and gas systems. To reduce the amount of ambient oxygen before production, the chamber and the melting system were recirculated with argon gas. The image of the gas atomization system used in the studies is shown in Figure 1.

Table 1. Chemical composition of Ni-Hard alloys.

Alloy Code	Ni-Hard 1	Ni-Hard 2	Ni-Hard 4
C	*2.8-3.6*	*2.4-3.0*	*2.5-3.6*
Si	0.8	0.8	≤ 2.0
Mn	≤ 2.0	≤ 2.0	≤ 2.0
S	≤ 0.15	≤ 0.15	≤ 0.15
P	≤ 0.30	≤ 0.30	≤ 0.10
Ni	3.3-5.0	3.3-5.0	4.5-7.0
Cr	1.4-4.0	1.4-4.0	7.0-11.0
Mo	≤1.0	≤1.0	≤1.5

Figure 1. Gas atomization system used during experimental studies.

The gas atomization system chamber is made of 304 quality stainless steel. After the raw materials were melted in the induction furnace, they were atomized with high pressure gases in the chamber. The induction melting furnace has a capacity of 3 kg. S type thermocouple was used to measure the temperature during melting. High-purity nitrogen gas was used to atomize the liquid metal. The atomization process was carried out at 35 bar by using a 0-50 bar pressure reducing regulator. A closed couple type nozzle was used during atomization processes. A ceramic melt delivery tube with a 4.5 mm hole diameter was selected to ensure liquid metal flow through the tundish. In order to ensure the continuity of the atomization process and prevent clogging, the pressure formation values at the end of the melt distribution tube were measured using a digital pressure difference meter (Testo 510i). The gas atomization parameters used in the study are given in Table 2. Within the scope of the study, firstly, high purity master alloy of Ni-Hard 4 alloy was prepared. During the preparation of the alloy, high purity pig iron and other alloying elements were melted separately.

Table 2. Atomization parameters.

Alloy Code	Ni-Hard 4
Melting Temperature (°C)	1200-1250(T_E) + 150
Gas Pressure (Bar)	35
Melt Delivery Tube (mm)	4.5
Gas Type	Nitrogen

The size distribution of the powders was carried out by sieve analysis. The images of the powders were taken using the JEOL 6060 scanning electron microscope. Afterwards, to be able to comment on the sphericity and fluidity of the powders, the obtained powders were subjected to the hall flow test, and then the angle of repose were measured and the results were shared.

3. RESULTS

Positive pressure values formed at the end of the melt delivery tube prevent the liquid metal from flowing through the pipe under the force of gravity, causing the liquid metal to be pushed back into the melt delivery tube and eventually clogging or damaging the melt delivery tube [10]. In order to ensure the continuity of the process before powder production with gas atomization, the aspiration pressure values formed at the end of the melt delivery tube were measured. The aspiration pressure graph is given in Figure 2.

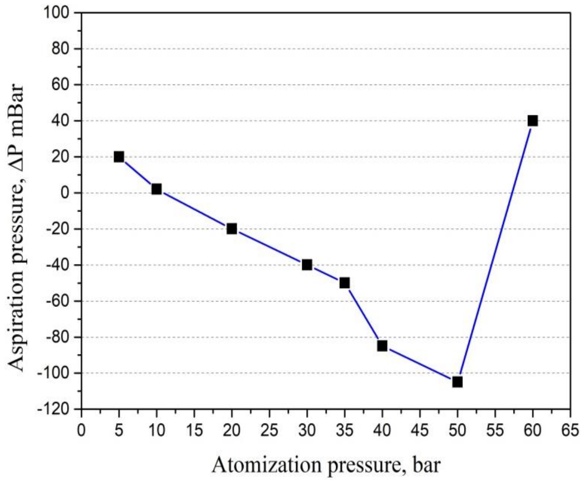

Figure 2. Aspiration pressure values at the outlet end of the melt delivery tube for different atomization working pressures.

After the measured aspiration pressures, the atomization pressure was determined as 35 bar to keep both the continuity of the atomization process and the (Gas/Metal Ratio) GMR optimal. This value was determined based on our previous studies [11-12]. Since the aspiration pressure is positive at atomization pressure values of 15 bar and below, it is predicted that the melt delivery tube will be clogged at the beginning of the process. As the negative aspiration pressure increases at 40 and 50 bar atomization pressures, the GMR will decrease. Therefore, the average particle sizes will increase. In their study, Urionabarrenetxea et al. atomized copper and copper alloys at different pressures. They revealed that increasing negative aspiration pressures decreased the GMR and therefore increased particle sizes [13].

SEM images of Ni-Hard powders are given in Figure 3. When the images are examined, it is seen that the Ni-Hard powders produced under 35 bar pressure mostly have a spherical shape, but long ligament particles draw attention in the image. Ligament structures are generally formed due to 2 parameters. Ligament structures appear due to insufficient kinetic energy transfer to the liquid metal in the primary atomization region due to high liquid metal viscosity and insufficient gas pressure [14].

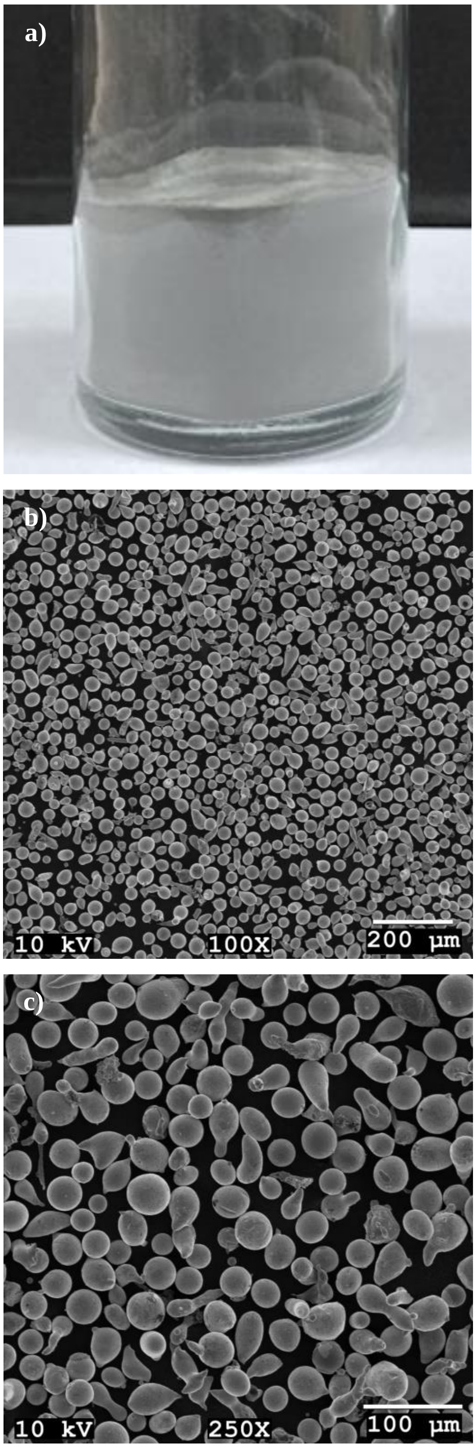

Figure 3. Macro and SEM images of Ni-Hard powders at different magnifications a) macro b) 100x c) 250x

The powders dimensions in the SEM images are in good agreement with the particle size distribution plot given in Figure 4. The average particle size of Ni-Hard 4 powders produced under 35 bar pressure was measured as (D_{50}) 31µm. Ni-Hard 4 powders exhibit a narrow size distribution range. For the cumulative distribution curve to move to the left, it is foreseen that the gas pressure should be increased. Qing et al. atomized the nickel-based superalloy under 1 2 3 4 and 5 MPa pressures, respectively. They showed that as the atomization pressure increases, the particle size distribution range will narrow and the highest particle size in the histogram will decrease. In short, they revealed that the average powder particle size will decrease [15].

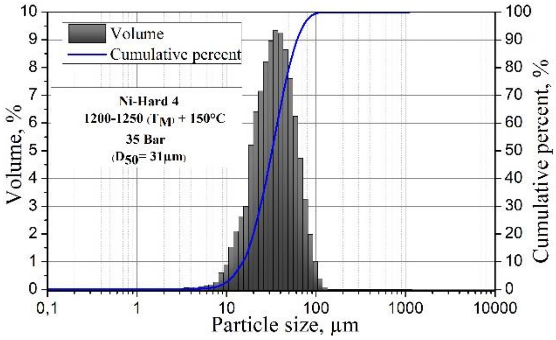

Figure 4. Particle size distribution graph of Ni-Hard 4 powders.

Since inert gases are used both during the melting process and during the atomization process, the surface of the powders is relatively smooth and no oxidations have been observed. Hall-Flow and angle of repose properties were determined for the fluidity properties. Figure 5 shows the measurement approach of the angle of repose and hall-flow properties. [16].

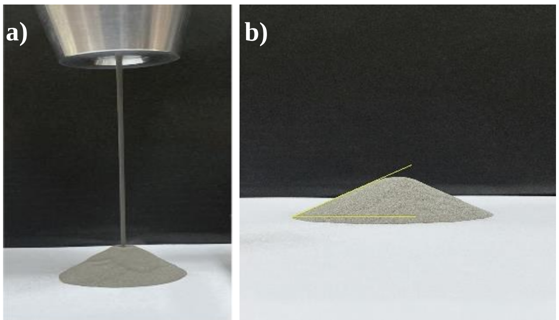

Figure 5. Hall flow measurement and angle of repose measurement a) Hall-flow b) angle of repose

The Hall flow rate test result of Ni-Hard powders was measured as 22 seconds for 50 g. In addition, the angle of the repose measurement result was determined as 28°. Zegzulka et al. in their study on metal powders with different shapes and particle sizes, revealed that powders with a standing angle of $20° < \alpha < 30°$ have free flow. They also emphasized that the angle of repose and the flow duration are directly proportional to the sphericity and impurity of the powders [17].

4. CONCLUSION

In this study, Ni-Hard 4 alloy was cast, and then Ni-Hard alloy powders were successfully produced by gas atomization technique. It has been shown that powders have mostly spherical shape due to the nature of gas atomization. In addition, the flow properties of Ni-Hard powders were investigated in detail. It was emphasized that spherical powders showed good fluidity. When the properties of the obtained powders were examined, it was revealed that they showed suitable characteristics for powder metallurgical processes. In recent years, the development of different alloys for the additive manufacturing sector has continued. Ni-Hard 4 alloy appears to be a potential alloy for the additive manufacturing sector, thanks to its superior mechanical and chemical properties (wear, hardness, corrosion resistance, etc.). When the powder properties obtained as a result of gas atomization are examined, additive manufacturing techniques can use this high-performance material.

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