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The evaluation of different environments in ultra-high frequency induction sintered powder metal compacts

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ABSTRACT: The application of the iron based Powder Metal (PM) compacts in Ultra-High Frequency Induction Sintering (UHFIS) was reviewed for different environments. The three different environments: atmosphere, argon and vacuum were applied to the PM compacts. Iron based PM compacts were sintered at 1120 °C for a total of 550 seconds by using induction sintering machines with 2.8 kW power and 900 kHz frequency. Micro structural properties, densities, roughness and micro hardness values were obtained for all environments. The results were compared with each other.

KEYWORDS: Induction; Iron; Powder metal compact; Sintering; Ultra-high frequency induction sintering

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RESUMEN: *Evaluación de diferentes ambientes en la sinterización de aleaciones pulvimetalúrgicas consolidadas por inducción de ultra-alta frecuencia*. Este trabajo comprende una revisión de la aplicación de la inducción de ultra-alta frecuencia (UHFIS) en la sinterización de aleaciones pulvimetalúrgicas de base hierro para diferentes ambientes. Los tres ambientes estudiados son: atmósfera, argón y vacío aplicados a material ya consolidado. Aleaciones base hierro ya compactadas se sinterizan a temperaturas de 1120 °C durante 550 segundos por medio de máquinas de sinterizado por inducción de potencia de 2,8 kW y 900 kHz de frecuencia. Se compararán las propiedades microestructurales, y los valores obtenidos de densidad, rugosidad y microdureza para todos los ambientes estudiados.

PALABRAS CLAVE: Consolidación de polvo de hierro; Hierro; Inducción; Sinterización; Sinterizado por inducción de ultra-alta frecuencia

1. INTRODUCTION

Powder metallurgy is a continually and rapidly evolving technology embracing most metallic and alloy materials, and a wide variety of forms. Powder metals is always a competitive technology compared to others noncommercial applications. The reasons for using powder metal technology include good tolerances, low cost, net shaping, high production rates, and controllable properties (German, 1994). In iron powder metallurgy, common sintering conditions are: 15–60 minutes at 1120–1150 °C (German, 2005). Particles of commercial iron powders for structural parts are usually ≤150 µm. Iron-copper-carbon alloys illustrate the unique properties possible by PM (German, 1994). Copper and graphite (carbon) are mixed with iron, and during sintering the copper forms a liquid phase. This gives the compacts better strength. Due to the fact that iron based compacts are used in a lot of industrial applications, iron based PM compacts were sintered by ultra-high frequency induction in this study. Many different types of models have been used in the powder metallurgical industry. Nowadays, most of the groups working in this field admit that metallic powders and green compacts have to be considered as granular materials. Plasticity models specially defined for geological materials are being applied on metallic aggregates (Riera and Prado, 2006).

In general, iron-based powder metal compacts are sintered by a conventional furnace and carried out at 1120 °C for 30 minutes under inert atmosphere (Çavdar and Atik, 2014a). Novel rapid sintering processes have emerged as an alternative to classical sintering methods. These are induction heating, microwave heating, plasma heating and laser heating methods (German, 1996).The induction sintering process is generally a hot consolidation technique of the PM compacts. This can be performed in a rigid die using uniaxial pressurization. The die is usually made from graphite to allow external induction heating (German, 1994). The most significant studies on sintering by induction are known as High Frequency Induction Heat Sintering (HFIHS). In the HFIHS process, high frequency (50 kHz) (Zinn and Semiatin, 1998) is used while the compacts are being pressed. Uniaxial hot pressing is slow and inherently has poor control over the heating and cooling stages because of the large thermal mass associated with the cooling. Typical maximum temperatures are 2200 °C and maximum pressures are 50 MPa. A vacuum is often selected for the process environment to minimize contamination of the compacts (German, 1994).

This method combines high-temperature exposure with pressure application over a short time. During HFIHS, a large current is induced in the sample and graphite die. As a result, the sample can be sintered uniformly and rapidly (Kim *et al.*, 2008). This sintering method is used in all kinds of different studies. Yang *et al.* (2008) sintered uranium oxide (UO_2) compacts at 1700 °C for 5 minutes using the HFIHS method. In this research they reported that when the heating rate and sample dimension were properly controlled, UO_2 pellets, with a density of more than 96% TD and an average grain size of ~6 μm could be produced within a few minutes. Relative density increased with increasing Fe_2O_3 content up to 0.5% mol and then decreased with further addition of Fe_2O_3, but grain size increased with increasing Fe_2O_3 content shown by Shon *et al.* (2009). Siemiaszko *et al.* (2013) investigated the influence of temperature during Pressure-Assisted Induction Sintering (PAIS) on the structure and properties of the Fe40Al intermetallic phase at the temperature between 720–1000 °C. In Shon *et al.* (2013) work, the synthesis of (Ti,Cr)C using high-energy ball milling and the sintering of (Ti,Cr)C was investigated using the HFIHS method which removed the need for binders. They produced a dense, fine-grained binderless (Ti,Cr)C hard material. Parka *et al.* (2014) studied TiO and Zr powders which were synthesized and densified from the high frequency induction heated sintering method within 4 minutes under high pressure (1 GPa) for biomedical applications. Relative densities of the sintered specimens between 98% and 100%. Kim *et al.* (2013a) presented the results of the sintering of (W,Ti)C and (W,Ti)C–NiAl3 composites by a high-frequency induction heated sintering with simultaneous application of induced current and high-pressure. They produced dense and nano crystalline (W,Ti)C and (W,T)C–NiAl3 hard materials in very short sintering times (<3 min). Kim *et al.* (2013b) investigated High-frequency induction heated sintering of High-energy ball milled TiC0.5N0.5 powders and the mechanical properties of the sintered products.

In previous studies, iron (Çavdar *et al.*, 2014a; Çavdar *et al.*, 2015) and iron-copper (Çavdar *et al.*, 2014b) compacts were cold isostatic pressed. Different kinds of iron based powder metal compacts were sintering by medium frequency induction (Çavdar and Atik, 2014a; Çavdar *et al.*, 2014b) or the conventional sintering method (Çavdar and Atik, 2014a; Çavdar and Atik, 2014b). Induction sintered compacts had higher density than conventional sintered compacts. Strength and micro hardness increased with increased sintering time (Çavdar *et al.*, 2015) and increasing copper content (up to 3%). It was reported that for medium frequency induction heated sintering process of iron-copper PM compacts, the optimum sintering temperature was at 1120 °C and sintering time was for 500 seconds (Çavdar and Atik, 2014a; Çavdar *et al.*, 2014a; Çavdar *et al.*, 2015). Çivi *et al.* (2014) sintered iron-copper PM compacts with the same induction system and investigated the reliability of mechanical properties. In addition to the experimental compacts, industrial PM compacts like bushings could also be sintering (Çavdar *et al.*, 2014b) by the medium frequency induction system.

For these HIFIHS studies, they used a 50 kHz induction generator and pressed compacts in the sintering process. However, we sintered iron and iron based powder metal compacts by a low-medium frequency (30–50 kHz) induction heated system after compacts were pressed by cold iso static pressing (Çavdar *et al.*, 2014b). In contrast to these studies, approximately 18 times higher frequency

(900 kHz) was used for the sintering process. In our previous studies, compacts were sintered in the continuous belt system, but in this work compacts were sintered in the middle of the induction coil and were stable. This UHFIW process can be also use for all materials. Not only the heating temperature can be calculated correctly but also temperature can be stabilized. Graphite molds helped to heat the sample and to attract the magnetic flux to the ceramic and non-magnetic materials in UHFIHS. In addition to the ultra-high frequency sintering process, the iron based PM compacts could be induction welded by the ultra-high frequency induction system (Çavdar and Gülşahin, 2014).

In addition to areas of usage of these induction systems, induction is used in hot forging processes. Çavdar (2014) investigated the effects of induction (250 kW, 3.2 kHz) and conventional heating of hot forged ANSI 1050 steel at 1230 °C and 1250 °C. Small grain sizes were observed in the microstructure of the induction heated specimens. In the hot forging process, surface qualities and colours of induction heated specimens were better than that of conventional heated specimens. It was observed that the induction heated process improved mechanical properties of steel samples in hot forging process. It was also observed that it produced more ductile and 7% harder specimens by using the induction system compared to the conventional furnace in the heating process of hot forging.

This paper presents the effects of the atmosphere, argon and vacuum environments in the ultra-high frequency induction sintering process for iron based powder metal compacts.

2. MATERIALS AND METHODS

The chemical composition of powders used in this work, which is used for commercial raw material gear production is given in Table 1. The powder compositions were compacted by Cold Isostatic Pressing (CIP) at 500 MPa in dimensions of Ø13 and 3 mm height. The PM compact was photographed with a 10 cent coin (Fig. 1) to show the real size of the compacts.

A horizontal coil was designed for the sintering process. The induction coil was constructed with a diameter of 4 mm and a wall thickness of 0.5 mm. The copper wire was wound once to produce a coil with an inner diameter of 20 mm. Magnetic flux was achieved homogenously by placing the PM compact in the center. The temperature of the sintering zone of the compacts was measured by an infrared thermometer (±5 °C) during the induction sintering process.

The PM compacts were sintered by a high frequency induction system at 1120 °C in 8.33 minutes (500 seconds) under three different environments: atmosphere, argon (10% H_2 90% N_2) in a 7 bar pressure and in a vacuum at 5 Pa. The vacuum and argon environments were applied to the sample in a 500×500×500 mm aluminum box. Front, side and back views of the system are shown in Figure 2 (a–c) respectively. Tempered glass was used in the front of the box for observing the process (Fig. 2a). A cable and vacuum accesses is shown in Figure 2b. In Figure 2c the gas entry value is shown. In general, inert gases or/and vacuum environments were used during the sintering processes. Static atmospheres or a vacuum protected the samples from the negative effects of air. Compacts were cooled naturally in the box under the applied environments.

Heating, sintering and cooling durations are given for the heat cycle of the UHFIS in Figure 3. The compacts were held at a temperature of 600 °C for 50 seconds during the pre-sintering process. The temperature of the compacts were then increased to 1120 °C over 100 seconds and sintered at this temperature 400 seconds. Finally the compacts were cooled naturally for 50 seconds. The total sintering process was 550 seconds.

Photograph images of the induction sintered PM compacts under the different environments argon, vacuum and atmospheric are given in Figure 4 (a–c) respectively.

The magnetic flux that interacts with the sample penetrates to a specific depth. This is defined as the "penetration depth" according to the literature (Siemiaszko *et al.*, 2013). Due to the ultra-high magnetic flux, the outer part of the compact was heated for a few second as seen in Figure 5. The red area, which is the nearest part to the induction coil, indicates the degree of magnetic flux penetration of the PM compacts. Penetration depth (expressed at cm) can be calculated using the equation (1), according to Zinn and Semiantin (1998):

$$d = 5000\sqrt{\rho / \mu f} \tag{1}$$

where ρ is a specific resistivity, μ is magnetic permeability and f is frequency. The sintering stage of each process is shown with a flow chart (Fig. 6).

The densities of the sintered samples were measured after the UHFIHS process using the Archimedes

Table 1. Chemical composition of green compacts

Composition	C	Cu	Ni	Mo	MnS	Amide Wax	Fe
wt. %	0.15–0.25	1.35–1.65	1.58–1.93	0.45–0.55	0.45–0.55	0.81–0.99	Rest

Figure 1. 10 cent coin and sintered PM compact image.

principle. Macro Brinell Hardness tests were carried out on the surface of sintered sample by using MMS 200 RB Macro Brinell Hardness Test equipment with a load of 100 kgf. Microstructure images and fractured surfaces of the samples were observed by using a JEOL JSM-6060 Scanning Electron Microscope.

3. RESULTS AND DISCUSSIONS

If we compare the surface appearance and sizes of the PM compacts, several significant differences were observed as follow. The PM compacts sintered in a vacuum were seen to have a bright surface appearance compared to those sintered in an atmospheric environment where a matt surface appearance was observed (Fig. 4b and c respectively). Sintering in different environments also caused variations in the size of the compacts. The largest size increase was due to the argon environments (Fig. 4a). This could be due to the diffusion of argon into the surface of the compact. The vacuum and atmospheric environments caused less change in size of the compacts, the least seen with the atmospheric environment. This is probably due to the oxidation of the surface of the PM compacts in the atmospheric environments.

Hardness measurements were applied to the surface of the compacts. Measurements points are given in Figure 7.

HRB hardness results are given in Table 2 based on the test point numbers in Figure 7. Due to the magnetic flux of the induction system, the hardest results were obtained from the hardness number point 3 in all processes. The same surface parts of the PM compacts were hardened during this sintering process. High frequency induction systems are actually used for surface hardening

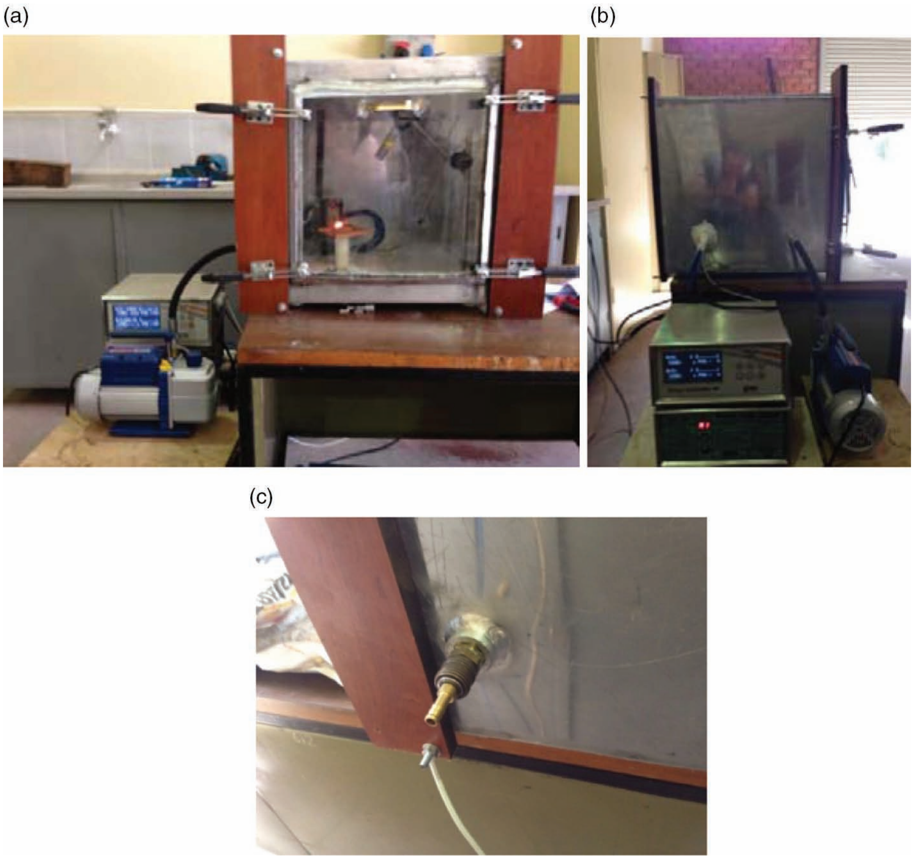

Figure 2. Photograph images of UHFIS system in a vacuum box: a) Front view, b) Side view and c) Back view.

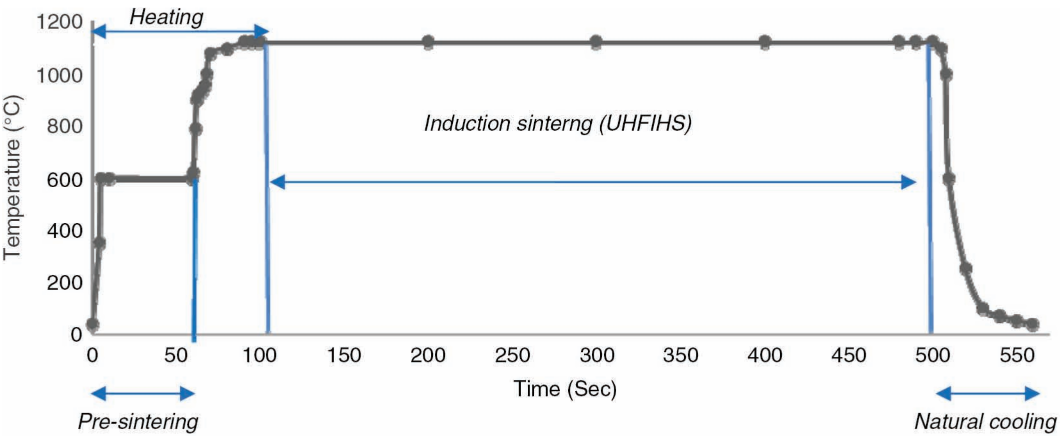

FIGURE 3. The heat cycle of the UHFIS process (* Error range is ±5 °C).

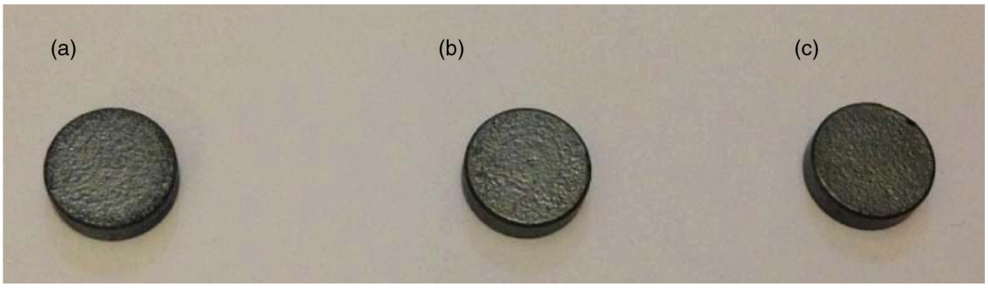

FIGURE 4. Ultra high frequency induction sintered PM compacts in different environments: a) Argon, b) Vacuum and c) Atmosphere.

operations of metals. Owing to the UHFIHS process, PM compacts are sintered and surface hardened at the same time.

Hardness test results taken from the surfaces of UHFIHS powder metal compacts at atmosphere environment are in agreement with the results of previous works (Çavdar *et al.*, 2014a; Çavdar *et al.*, 2014b) which were around 50 HRB. In addition to these hardness results, conventional sintered iron based PM compacts' hardness (Çavdar and Gülşahin, 2014) was almost 46 HRB. Both argon and vacuum environments improved the average surface hardness of the PM compacts.

The average density results observed were; 7.2 g cm^{-3}, 7.1 g cm^{-3} and 6.9 g cm^{-3} from argon, vacuum and atmosphere environments respectively. The error range of all densities was between ±1%. As compared to the density results of previous studies when iron based PM compacts were sintered by the medium frequency (30–50 kHz) induction system (Çavdar and Atik, 2014a; Çavdar and Atik, 2014b; Çavdar *et al.*, 2014b) at atmosphere environment and UHFIHS powder metal compacts at the same environment, densities of previous studies were almost the same at around 6.8 g cm^{-3}. The highest density results were obtained from ultra-high frequency induction sintered compacts in the argon environment. Unexpectedly, the density result of sintered compacts in the argon environment was only a little higher than the sintered compacts in the vacuum environments. It was immediately obvious that 5 Pa vacuum was not enough for this system. With the appropriate vacuums, both sintered processes with the vacuum and argon environments improved the density results of the PM compacts compared to the medium and high frequency induction sintered iron based PM compacts densities with the atmosphere environment.

Microstructure surface images of the PM compacts observed are shown in Figure 8. Microstructure images (Fig. 8) were taken from the cross-section of the iron based compact which were gridded, polished and etched (2% NH_3, 98% alcohol).

Microstructure images of the PM compacts which were given in Figure 8 (a, c and e) were taken from near the cross-section's outer surface. The other images, Figure 8 (b, d, and f), were taken from the inner region of the PM compacts. These images represent the internal images of the compacts. It can be clearly seen that there are changes in the microstructures of the surface and inner regions for the different environments (Fig. 8a, c and e). This could

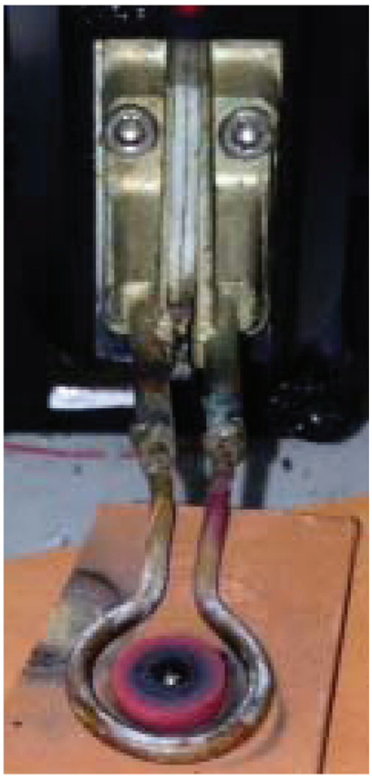

FIGURE 5. Heating PM compacts at 3 seconds.

be due to the magnetic field of the induction system or due to the environments. These images show that three different surfaces were obtained for each environment. These changes were measured from the microstructure image (Fig. 8a, c and e) and the average of the results for different sintering environments are given in Table 3. It is obvious that the microstructure changes are very different for each environment. The depth of change with the argon and atmosphere environments being around 3 times more than with the vacuum environments. This proves that there is an improvement in the surface

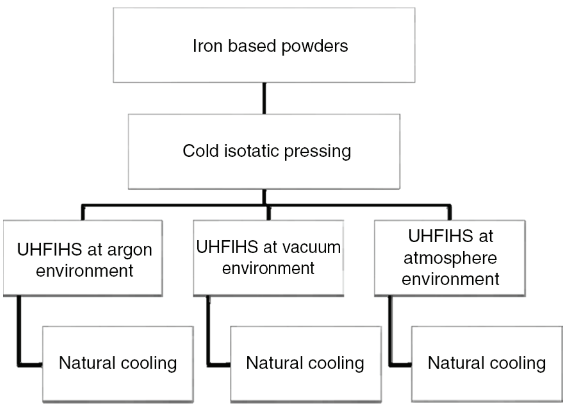

FIGURE 6. Flow chart of all sintering and sintering processes.

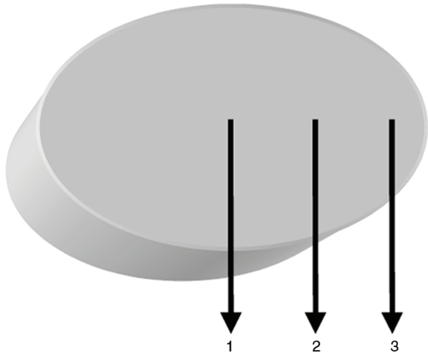

FIGURE 7. Hardness test points of the PM compacts.

and structure while PM or nano compacts are sintered using a vacuum environment. Surprisingly, the highest depth of change measurement was seen under the argon environment. The applied argon gas to the sintering process causes alterations to the chemical composition of the iron based PM compacts. This is also seen with nitruration, cementation and decarburization heat treatments. This chemical alteration causes the highest depth of change measurement.

The microstructure images given in Figure 8 (d and f) are very similar. Similar phases were seen in both images. They demonstrate that in the sintering process, inert atmosphere (argon) and atmosphere environments, some different phases occurred compared to the vacuum (Fig. 8b) environment in the inner region of the PM compacts. This proves that the vacuum environments also improved the inner region of the microstructure of the PM compacts too.

A degree of argon gas diffusion was observed as seen in Figure 8c. This is the grey area at the bottom of the image. The decarburization zone can be seen from

TABLE 2. Average results of hardness of the induction sintered compacts

Hardness [HRB]*	**1**	**2**	**3**
At argon environment	50	55	58
At vacuum environment	50	53	55
At atmosphere environment	49	50	52

*Error range is between ±1%.

TABLE 3. Changes from surface to center for the different sintering atmospheres

Sintering environments	**Depth of change (mm)**
Argon	444
Vacuum	110
Atmosphere	376

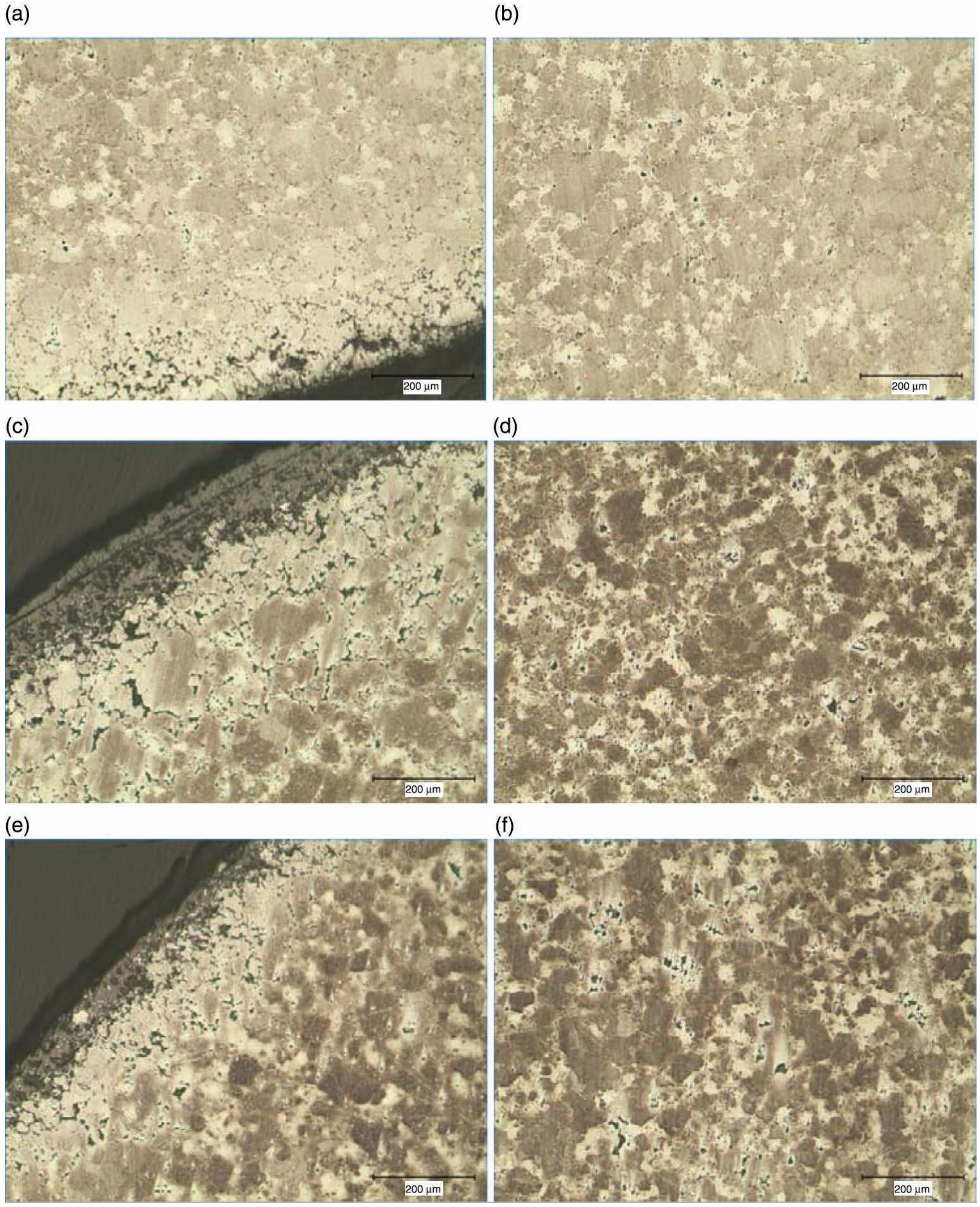

FIGURE 8. Microstructure images of the UHFIHS PM compact: a) and b) in the vacuum environment (×100), c) and d) in the argon environment (×100), e) and f) in the atmosphere environment (×100).

surface to the center of the cross-section in microstructure (Fig. 8e). In the bottom of the image the brighter zones reflect the decarburization zone. However, an oxide layer formation was observed in the same image. No decarburization zone or oxide layer were observed as seen in Figure 8a. This evidence explains how the vacuum environment play an important role in the sintering process of the powder compacts.

Average roughness (Ra) and maximum peak (Ry) values were measured from the surfaces of the PM compacts sintered with three different sintering environments. Those roughness results are given in Table 4.

According to the roughness results, the highest peak (Ry) and average roughness measurements (Ra) were seen in the PM compacts sintered in the atmosphere environment. The argon environment

TABLE 4. The roughness results for the different sintering atmospheres

Sintering environments	Ra (µm)	Ry (µm)
Argon	3.29	23.14
Vacuum	3.43	26.32
Atmosphere	3.93	29.27

and vacuum environments improved the roughness values of the PM compacts as can be seen in Table 4. The argon environment gives the most improved surface roughness result. It illustrated that the vacuum pressure was not enough for iron based compacts.

The penetration depth was 0.7 mm when we used an induction generator with a power of 2.8 kW and frequency of 900 kHz. Same penetration depth was calculated by Cavdar and Gülşahin (2014) welding application of the PM compacts. This is the induction hardened depth of the induction sintered region. It was calculated that, 13.26 kW.h^{-1} of energy was needed for ultra-high frequency induction sintering of 1 kg of PM. In addition to this energy calculation, Çavdar and Atik (2014b) reported that 82 kW.h^{-1} of energy had to be spent for sintering 1 kg of PM compact in conventional sintering. But using the induction sintering method, it was calculated that only 24.49 kW.h^{-1} was spent for the same PM compact.

Due to the need for homogenous heating of the compacts, induction coils were made according to the samples shape. Induction coil design was studied for the medium frequency induction sintering process and high frequency induction sintering in previous works. In the next studies, the vacuum atmosphere will be upgraded for UHFIS process of nano and powder materials.

4. CONCLUSIONS

The following results were obtained for Ultra-High Frequency Induction Heated Sintered (UHFIHS) iron based powder metal compacts in different sintering environments:

- Iron based PM compacts were sintered successfully with the ultra-high frequency induction system.
- Both argon and vacuum environments improved the densities, roughness and micro hardness results of the iron based PM compacts.
- Using a ultra-high frequency induction system in the sintering process, allowed compacts to be sintered and hardened at the same time.

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