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Effect of Microwave and Field Assisted Sintering on the Mechanical Properties of Inconel 718 MIM Samples

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Abstract
In this investigation, three different ways of sintering Inconel 718 MIM samples are compared. The conventional way of sintering in a furnace will be compared to FAHP and microwave sintering. The difficulty of these two methods is to be able to control the shrinkage of the sample and so its shape. The sintered samples were all injected from a feedstock composed of a fine particle Inconel powder and a binder principally composed of CAB (cellulose acetate butyrate) and PEG (Polyethylene Glycol). They were debinded in water and put in a furnace. After the application of the different sintering processes, the effects on the microstructures and the mechanical properties are compared. There was no difference in distribution of pores between the conventional sintering and the FAHP sintering but FAHP samples showed better hardness. The microwave sintering of a MIM sample is more complex and the best properties were not obtained.

Introduction

Nickel Chrome super alloys exhibit good mechanical strength and high resistance to creep at high temperatures in addition to corrosion resistance [1]. Thus, they can be operated in combined conditions of strength and temperature under the influence of a corrosive media. The problem of this kind of material is the difficulty to machine a mechanical part due to its ductility, imposing dimensional limits and strong loss of material in order to obtain the desired component [2]. Metal Injection Molding (MIM) relies on shaping metal particles, and then sintering them. This method combines the advantages of PM and the flexibility of the thermoplastic molding process. The final product is nearly full density and with mechanical characteristics close to the machined [3]. The high production quantities of complex shaped components coupled with low loss of material makes this method competitive with the other methods [4]. There are three steps in the MIM process: the formulation of a feedstock by mixing of a metallic powder with a polymeric binder, the molding of that feedstock by injection into tooling with a determined pressure, and finally the solvent and thermal processing of the shaped sample. In the case of Inconel 718, the sintering is followed by an aging treatment in order to obtain the best performance of the alloy [5]. This method consumes a lot of time and power. In order to improve these parameters, new ways of sintering have been investigated. The Field assisted hot pressing (FAHP) is a method inspired by conventional hot pressing. The powder is introduced into a graphite die (punches and matrices) and submitted under pressure and crossed by an electric current of high intensity. The use of a uni-axial pressure combined with the electric pulses accelerates the sintering kinetics, hence obtaining in a short period of time near fully dense components [6]. In this way, it is also limiting the grain growth and so, the development of fine microstructures bringing sometimes to better performances [7]. The microwave sintering has been used essentially on ceramics and composites materials since the beginning of the 70's. It was established that the heating of the sample was induced by the movement of the charges particles inside the materials (ions, dipoles) created by the microwaves [8]. The rate of the heating depends on the penetration of the microwave electric field. Janney et al. [9] sintered alumina by this method and showed an improvement of the kinetic of sintering and a reduction of the sintering temperature of 300°C. The microwaves sintering is a process appreciated for industrial applications because it allows reducing the sintering time and the energy used. Unfortunately, the metallic materials are reflecting most of the microwaves, this is why during a long period of time and this domain was investigated only recently [10]. In the case of a metallic powder, it was proven that the microwaves were able to penetrate the material by the porosities [11]. By increasing the heating rate, this method has also an effect on the grain size distribution and so the mechanical properties [12].This study is based on the comparison between conventional, FAHP and microwave sintering of MIM samples. There is no information about the processing of Inconel 718 through these two new methods. The first step is to optimize the sintering parameters of these two methods by finding the best density while keeping the advantages of

the MIM process with complex geometry. The microstructures in function of the sintering temperature will allow comparing the behavior of the material at different heating rates. The effect of this parameter directly impacts the grain size and so the mechanical properties, so the hardness of the samples is compared.

2. Material and Methods

2.1 Powder and binder

The Inconel 718 powder is provided by Sandvik Osprey Ltd and its chemical composition is presented in Table 1. The particle size distribution has been measured (figure 1) and the diameter values corresponded to cumulative volume fractions D_{10}, D_{50} and D_{90} are 3.53 µm, 6.24 µm and 10.97 µm respectively. The standard deviation is 0.86 µm. According to the literature [13], the structure of the Inconel 718 after sintering is composed by a γ matrix reinforced by a γ' phase Ni_3 (Al, Ti) and a γ'' phase Ni_3 (Nb, Ti). These two intermetallic phases are responsible for a rise of the hardness and the good performance of the alloy at high temperature. The presence of carbon, that can bring the formation of Niobium and Titanium carbides, must be limited as there is a probability of lowering the mechanical performance of the component.

Table1: Chemical composition in mass percentage of the Inconel 718 powder

Ni	Fe	Cr	Mo	Al	Ti	Nb	C
52.5	19.0	19.0	3.0	0.5	0.9	5.3	0.0

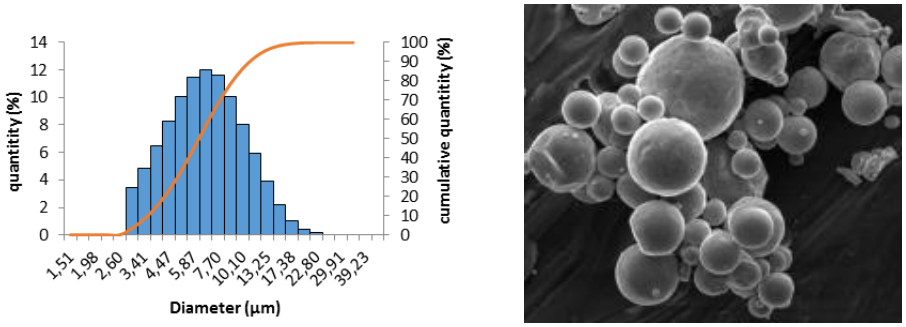

Figure 1 Particles size distribution and MEB picture of the Inconel 718 powder

The selected binder is composed of cellulose acetate butyrate (CAB 30K), Polyethylene Glycol (PEG 4K and 20K) Stearic Acid (SA) and Phenothiazine (PTZ). This type of binder was selected due to its ecological features because PEG can be eliminated by water and CAB comes from a cellulosic natural raw source. The compatibility of this binder with fine size powder is also helping during the injection [14]. PEG is used to improve the flow of the mix and make the injection easier. CAB's purpose is to rigidify the green part structure and allowing manipulation after debinding. SA is a long chain polymer which assures some homogeneity inside the feedstock.The green parts undergo a first debinding step. They are introduced into distilled water at room temperature for 48 hours in order to get rid of the PEG. They are then dried in an oven at 50°C for 5 hours. The CAB is eliminated by thermal debinding at 500°C for 3 hours under Argon atmosphere. A heating temperature rate of 2°C/ min is applied during all the cycle in order to avoid overpressure inside the sample during the degradation of the polymer.

2.2 Conventional sintering

The MIM samples are sintered in a high vacuum furnace. A heating temperature rate of 5°C/min is applied during all the cycle. The sample is maintained at 1290°C during 2 hours in order to reach the higher level of density [15]. The sample is then cooled down inside the oven after 2 hours. The density of the samples is then measured by the use of Archimedes' method. They are also ground and polished to a mirror-like surfaces, using colloidal silica at the final stage, in order to observe the microstructure obtained on a microscope Nikon Eclipse-LV150 and particle distribution. The hardness is finally measured by a Vickers test on a Shimadzu HMV with a load of 1.0 HV with at least 3 measurements at the centre and at the edge of the sample.

2.3 FAHP sintering

In the case of the FAHP, the Inconel 718 powder is directly introduced into a graphite die. The graphite die is composed of main cylinder pierced with an internal diameter of 20 mm and of two punches of the same diameter. In order to protect the equipment and to improve the electric contacts between the different parts of the system, a foil is placed between the powder and the die. The foil is composed of pure tungsten, which will block the carbon diffusion inside the Inconel 718. If the powder is in contact with carbon during the test, an important diffusion of the carbon could weaken the characteristics of the sample [16]. The samples have been consolidated in a Gleeble 3800 equipment by the application of an alternating current of 50Hz going through the punches under vacuum. The level of current is adjusted automatically by adjusting the temperature of the samples measured via two thermocouples and the programmed temperature. One is at the center of one of the punches and the second one close to the sample inside the graphite die. The shrinkage of the sample is measured via the displacement of the punches. The heating rate of the sample is fixed at 50°C/min until reaching the desired temperature. A dwell time is applied at 600°C during the application of a minimum pressure of 5 MPa and it is maintained until the cooling is done.

2.4 Microwave sintering

The samples are sintered inside a microwave furnace provided by Sairem. The sample is introduced inside the chamber composed of a quartz tube and positioned thanks to an alumina tube in front of the microwave source. The control of the atmosphere inside the chamber is done via a vacuum pump and different inlets of gas. In our case, Argon is going to be used thanks to its facility to be ionized [17]. The power of the microwaves can be set between 0.10 and 3.0 kW. The microwave source allows generating plasma inside the quartz tube which serves as a dielectric. The electric field of the microwaves is propagating between the tube and a waveguide providing an electric field absorbed by the plasma. This principle allows the creation and the hold up a plasma column of a certain length, depending of the pressure, the power and the nature of the gas. Depending on the gas used, a plasma flame can go to high temperature at a really fast rate [18]. This equipment was designed to hold up until around 2300°C. The heating rate and the temperature obtained during sintering depend on the length of the flame and the distance from the sample. The power of the microwave source is increased step by step by 100°C every minute until around 1300°C during a measured time. The power presented in the results is the real power that was absorbed by the plasma flame and the sample. It is obtained by the subtraction of the power sent by the source and the measured power that is going back to the source.

3. Results and discussion

3.1 Conventional sintering of Inconel 718 MIM sample

The evolution of the shrinkage in Figure 2 of the MIM cylinders during the sintering. The porosity of the sample can be observed between the particles that could come from the debinding of the polymers with water. There is two decrease of the height of the sample, a first one around 160°C corresponding to the thermal debinding of the CAB and the second starts around 1090°C showing the beginning of the sintering.

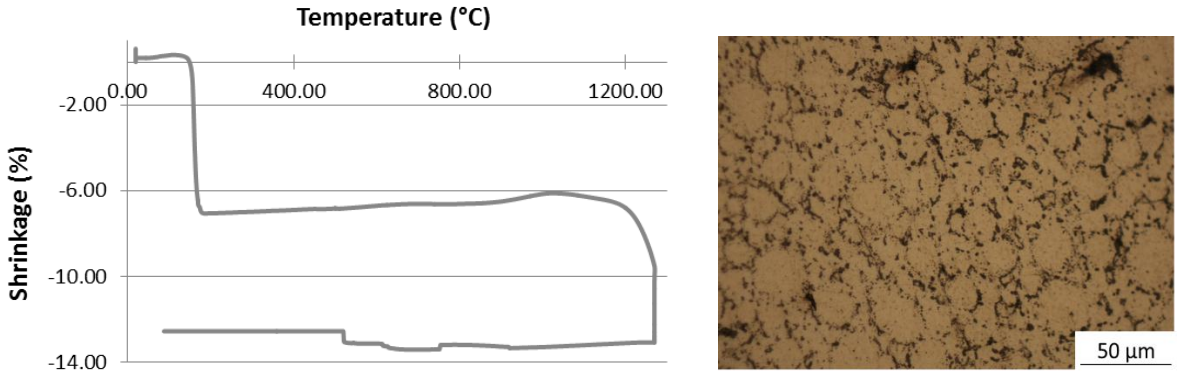

Figure 2 Dilatometric curve of Inconel 718 sintered at 1290°C during 2 hours at 1°C/min

3.2 Behavior of Inconel 718 during FAHP sintering

After the debinding step, the MIM sample is only composed of powder and a little bit of polymer barely holding the particles. Because of that, they are extremely brittle and cannot be properly manipulated. In order to be able to use them in the Gleeble equipment, the samples are pre-sintered at 700°C during 1 hour, in order to start the consolidation while limiting the effects on the Inconel γ phase.A temperature of 1290°C under conventional sintering conditions is necessary for the consolidation of Inconel 718 powder. The application of a pressure during FAHP sintering brings additional energy to the system, lowering the sintering temperature of the material. Three different temperatures have been tested in order to obtain a high density.

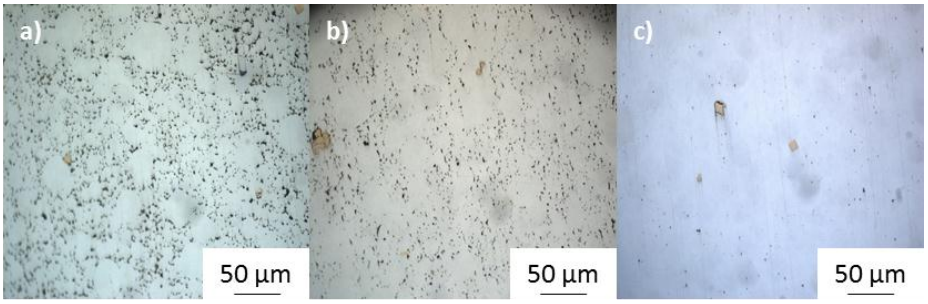

Figure 3 Optical Microscopy images of the samples sintered by FAHP at a)1100°C b)1200°C and c) 1250°C

The best density was obtained with a temperature of 1250°C, reaching 94.5% of the theoretical density. At 1250°C, the porosity can still be observed but a well-developed microstructure can be seen. At this temperature, the measured shrinkage has been measured and it was not improved at a higher temperature.

3.4 Microwave sintering of MIM sample

The microstructures of the two samples presented in figure 5 were obtained after a sintering time of 30 and 60 min. After 30 min, the powder particles size distribution observable varies between 20 and 40 µm. The growth behavior of the particles looks similar to the conventional way at a lower temperature. However, the ratio between large and small particles is not the same and could be explained by a phenomenon already observed with other materials. It has been observed the formation of hot points on some samples followed by an abnormal enlargement of the particles. The formations of these points are explained by reaching locally high temperatures and after a critical temperature, the particles starts to sinter before the others, resulting in an inhomogeneous growth of the particles during sintering. A too high power of the microwaves applied on the material explains this phenomenon [17]. After 1 hour of sintering, the microstructure looks more homogenous and no more particles are observable. Some cracks can be observed on the surface. They could be explained by the debinding step when the heating rate was too high and damaged the shape of the sample.

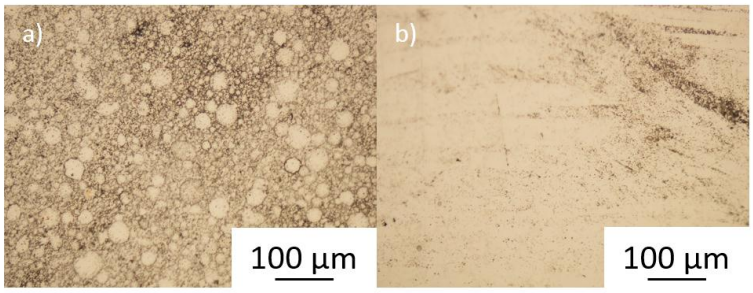

Figure 4 Optical Microscopy images of the samples sintered by microwave sintering during a) 30min and b) 60min

During the microwave sintering, the evolution of the temperature and the power absorbed by the system were measured. When the sintering temperature was reached, the power needed to keep a constant temperature remained constant. After some minutes, more power was needed until reaching the limit of 3kW of the equipment. At the end of the 1 hour sintering time, it was not enough maintain

this temperature. This effect could be explained by the closing of the porosities with the time. Because of the lack of connection between the porosities, it was harder for the microwaves to go through the sample and more power was needed to force the way [18-19].

4. Conclusions

The density of the MIM sample sintered conventionally is the highest of the three methods. The FAHP sintering is giving the lowest density because of the obligation to use a low pressure during the test. There is a possibility to improve it by working on the sintering heat rate and dwelling time. On the other hand, the FAHP method is giving the best results in terms of Hardness. A fast heating rate and a low sintering time is limiting the particle growth. According to the Hall-Petch effect, it is implying better yield stress characteristics. The microwave sintering is showing the lower hardness. Two factors can explain this result, the first being the cracks observed on the surface because of the brutal debinding, but also because of the hot points formed inside the sample during the sintering. In terms of kinetics, the faster method is the FAHP. The sintering time of the Inconel is cut by half between the microwave sintering and the conventional sintering.

Table 2 Comparison of the methods employed and the properties obtained

Method	Conventional	Micro-wave	FAHP
Temperature	1290°C	1300°C	1250°C
Dwell time	120 min	60 min	10 min
density	96.70%	95.8%	94.50%
hardness	211.3 ± 3.4	191.2 ± 15.8	256.3 ± 5.7

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