THE INFLUENCE OF SINTERING AND NITRIDING PROCESSES ON DISTALOY AE POWDER MATERIALS

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ABSTRACT

In this work, Distaloy AE metal powders have been chosen for study due to widely used in industrial applications. First, Distaloy AE powders were pressed at 500 MPa pressure and room temperature. Then, pressed specimens were sintered at 1120 °C temperature for 30 minutes. In order to examine the effects of sintering operation on density, densities of each specimen have been measured separately. After sintering operation nitriding was applied to a group of specimen at 520 °C temperature during 16 hours. To investigate mechanical properties of sintered and nitriding applied Distaloy AE powders, tensile and fatigue tests were performed. In addition, fractured specimens after tensile tests were used for micro-hardness tests. For metallographic researches of specimens, optic microscope and scanning electron microscope test were examined.

Key Words: Distaloy AE, Sintering, Nitriding, Fatigue

ÖZET


Anahtar Kelimeler: Distaloy AE, Sinterleme, Nitürleme, Yorulma

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Introduction

The Powder Metallurgy (PM) process is a near-net or net-shape manufacturing process that combines the features of shape-making technology for powder compaction with the development of final material and design properties (physical and mechanical) during subsequent densification or consolidation processes (e.g., sintering). It is critical to recognize this interrelationship at the outset of the design process because a subtle change in the manufacturing process can cause a significant change in material properties. Powder metallurgy has a wide range of applications ranging from automotive, building and contraction, hardware, heat treatment, medical and dental, to advanced aerospace components, etc. PM components have established themselves as an economic alternative to components made from other manufacturing processes as well as the only means to procedure some components which cannot be made other methods (Angelo and Subramanian, 2009).

This process is a very suitable technique for manufacturing. Especially, this process contains blending or mixing, pressing or compacting, sintering and finishing [10]. An important step is sintering for this process. Sintering reduces the porosity and enhances properties such as strength, translucency and thermal conductivity; yet, in other cases, it may be useful to increase its strength but keeping its gas absorbency constant. During the firing process and as it continues; grain size becomes smaller and more spherical.

The most important thing after producing the material by using Powder metallurgy is to increase the mechanical and physical properties of the material. This especially can be done by nitriding, carbid ing. Nitriding is low temperature, low distortion "thermochemical" heat treatment carried out to enhance the surface properties of finished or near finished ferrous components. It is different in terms of suitable materials, processing conditions, the nature of the surface layers imparted and the property improvements conferred. Nitriding, conducted in gas (490°C -560°C) or plasma (400°C - 590°C) for treatment times ranging up to 90 hours, involves the diffusion of nitrogen into the surface to produce a controlled depth of hard alloy-nitrides. Unlike the high-temperature, case-hardening treatments, hardening is achieved without the need for quenching (Kang, 2005).

Nitriding process has some advantages to improve qualified for samples, shown in Figure 1. These are high torque, high wear resistance, abrasive wear resistance, corrosion resistance and high surface compressive strength (Pye, 2003).

This paper presents the influence of sintering and nitriding process on Distaloy AE PM. The main aim of these processes is to improve physical and chemical characteristic of Distaloy AE. To do this, one should know the properties of the material well, adjust the temperature of the thermal process, choose a process increasing the surface quality and suitable for sample and perform a good fatigue design.
Material and Method

Material

Distaloy AE powder is one of the diffusion alloy of the most common heat-treatable grades and used in this study. The Distaloy AE based on additions of iron (Fe), nickel (Ni), copper (Cu), molybdenum (Mo) and carbon (C) is a high strength alloy. High Ni content and good compressibility makes it possible to produce materials with a sintered tensile strength increased. Distaloy AE exhibits good hardenability and dimensional stability (Rajan et al, 1994).

Material used in this study was Distaloy AE powder from Höganas in Sweden. The chemical composition of Distaloy AE powder is given in Table 1.

Table 1. Chemical analysis of Distaloy AE powder

<table>
<thead>
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<th>Chemical Analysis of Distaloy AE %</th>
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<tr>
<td>Iron (Fe)</td>
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<td>94</td>
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Method

First of all, the components of Distaloy AE powder (4% Ni, 1.5% Cu, 0.5% Mo, 0.01 C); which are approximately weight of 37 g are mixed to put into blanks which a die in sizes of 10x10x55 mm³, together with lubricant, until a homogeneous mix is obtained be pressed under 500 MPa pressure at room temperature. Then, the specimens were sintered under 1120 °C sintering was selected the best temperature to produce the samples for tensile and fatigue tests since it resulted in high density [19]. All specimens of pressed dies were sintered for 30 minutes at 1120 °C temperature in each sample, respectively. Temperature was increased by 5 °C/min until medium of furnace reached sintering temperature; and then specimens were kept for 1 hour. After, furnace was closed and temperature was decreased by 5 °C/min, until medium of furnace was reached room temperature (RT, 22°C). After this process, density of sintered and green Distaloy AE was evaluated by the Archimedes Principle. After, the sintered samples were conducted machining operations and polishing operations using Computerized Numerical Control (CNC) turning lathe and metal polishing machine, in turn, to achieve optimum results for fatigue and tensile test produced. After that, fatigue and tensile test samples and technical drawings are shown in Figure 1 and 2 separately.
In this study, nitriding was applied to increase fatigue life of material. To evaluate sintered and nitrided parts in respect to mechanical properties. 11 samples were only sintered and other 11 samples are nitrided at 520 °C temperature and 16 hours. in % 50 H2 and % 50 N2 atmosphere. After this process, tensile and fatigue test samples were prepared.

All the experiments of fatigue tests were conducted in the axial load control which is more severe than bending under fully reversed loading (stress or load) ratio R = σ min/ σ max=-1 which is sine (cyclic) waveforms using 11 specimens for the determination of each S-N (Wöhler) curve. The fatigue tests were performed at the constant frequency of f = 10Hz. One stress ratios (R = -1) were investigated on the unnotched (Kt=1) specimens. Sine (cyclic) waveforms are used. The cyclic group of waveforms all start at a setpoint, proceed to a peak amplitude, either negative or positive, before reversing direction, passing through the setpoint to the opposite peak amplitude. The signal will then return to the initial starting position, ready to commence the next cycle. The sine waveform follows a sinusoidal path, defined by the amplitude and frequency parameters. In the tensile test, experiment was taken v = 0, 5 mm/min as vertical movement and this velocity was kept constant during experiments. Averages of these tests were taken and besides, graph of the results were evaluated in different colors and type for each test.

Furthermore, the vickers hardness profile of fractured parts obtained as a result of static tests was measured at 14 different points and intervals of 3 mm on longitudinal section using a Vickers intender with a 25 g load for time of 10 s as displayed in Figure 3.
In order to analyze the morphology and microstructure of materials which were sintered and nitriding in different conditions, specimens were examined by Optical Microscopy (OM) and Scanning Electron Microscopy (SEM). Optical microscopy has five main steps for preparation of specimen in this study, shown in Figure 4.

For this reason, the significant rupture surfaces of materials were prepared by standard metallographic techniques unetched and etched with reagent in order to study the grain structure and allow microscopy characterization of sintered and nitriding materials.

**Result and discussion**

In this study, green densities of Distaloy AE powders were determined as 7.19 g/cm³ by evaluating the average of eleven specimens pressed at 500 MPa. The average densities of sintered and nitreded samples were found as 7.13 g/cm³ and 7.15 g/cm³ respectively. As can be seen from this, green density of samples was higher than sintered density of samples. The reason for this, the decrease in number of pores and becoming the internal structure of samples more
homogeneous. Hardness extremely increased after nitriding as can be easily and clearly understood from the graph shown Figure 5. The main reason for this, the occurrence of a layer above the nitrided sample as seen in Figure 6. Nitriding layer of samples

Figure 5. Compare hardness of sintered and nitrided specimens

Figure 6. Nitriding layer of samples

This layer provides the surface of the material gets more severe and as a result be resistant against impacts. The hardness increases by 163 percent on the surface and by 39 percent in the inner sides as can be seen from the graph.
The result of tensile test about sintered and nitride specimens shown in Figure 7. It is possible to make a comment about the tensile stress of samples as seen from the graph. Sintered sample ruptured 297 MPa of maximum tensile stress while the nitried one had 318 MPa tensile stress of maximum. Diffusion of nitride atoms through the sample plays an important role for making is stronger. Nitride atoms enter the gaps and pores of the sample and makes its tensile stress increase by 7 percent.

![Figure 7. Comparison sintered and nitried specimens of tensile stress curves](image)

In fatigue experiments, 50-140 MPa as a stress value of average are used and graphs are generated. $2 \times 10^6$ value was accepted as a limit in the graph as previous ones and above this value was run-out period. After nitriding, nominal stress increased by 15-20 % and the lifetime of sample was better. 90 MPa stress, sintered sample ruptured after 379000 cycle while the nitried one ruptured after 1513601. The main effect on this condition is the diffusion of nitrite atoms through the inner side of the sample and fill the gaps and pores. After that, the inner side of the sample is becoming more stable and homogenous, and so that the lifespan gets longer shown Figure 8.
The properties, distribution, porosity, fractures, residues of the phases in the material are seen in the optical microscope. Figure 9a to only sintered samples and Figure 9b to nitrided ones. After pressing method, the macro-structure cannot be homogenous completely thus the sintered samples especially have high porosity. In Figure 9a macro pores in the sample can be seen. These pores make the lifetime of the sample shorter and the sample more brittle. The amount of the macro pores is less in nitrided samples compared to the sintered ones shown in Figure 9b. This is because of the diffusion of the nitride atoms through the inner side of the sample and make it more homogenous and so a higher resistance.

In pictures, serrate (S), ductile (D), cleavage (C) are expressed. Figure 9a sintered samples. Ductiles and cleavage can be easily seen in this figure. A big space and fracture can be easily seen. Samples used in the experimental study.
have a different porosity value and distribution in the border and center line and also those regions would have a different damages under applied load. In Figure 11, spaces and serrate due to fatigue and at the edge of the material can be easily seen. The reason why the spaces occur is the effect of the load on material. In Figure 11, the fracture occured over the surface of the material, can be seen, and is progressing from the edge surface to the inner and heterogenous distributed pores so causes damage. Chappies and pores seen in figure have a homogenous distribution and are at the edge of the material.

Figure 11. SEM views of fracture surface of sintered(a) and nitreded(b) specimen at 100 MPa fatigue; X500

Conclusion
In this study Distaloy AE powder is used successfully in the experiments. In these experiments, sintered and nitrited samples are used and obtained values are transferred to tables and graphs. Values obtained from experimental studies can be sequestered as below

The density of Distaloy AE powder was measured in two different conditions and nitrited sample density (7.12 at room temperature) was more than sintered one. After the Tensile test, the resistance of the sample increased more in nitriding, the Ultimate Tensile Strength was 8 percent more than sintering. Fatigue resistance is 50-120 MPa for sintered samples and 70-140 MPa for nitrided samples. At 100 MPa pressure, sintered sample ruptured 120000 rpm, nitrided sample ruptured at 150000 rpm. The increase in the density of the nitrided sample was simply because of the decrease in pore amount.

When we compared the hardness of the samples, nitrided ones had stiff film layer on the surface thus was harder. The hardness value on the surface is 450 HV, and while getting through the inner side it decreases. Inner side of both sintered and nitrided samples are homogenous but the pore amount of nitrided
samples is less. The reason why the nitrided ones are more stable is the pore shrinkage.

In SEM, we can easily see the surfaces of the cracked samples. Fractures, pores, chappies (which are less in nitrided samples compared to sintered samples) occurred over the surface is due to the fatigue duration.

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