Investigation of the Synthesizing Effects of Prealloyed NiTi + Pure Al(2, 4, 6, 8 wt.%) Powders by MA and Sintering

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Abstract: Prealloyed NiTi and high purity Al powders were used in the study. Prealloyed NiTi powders and Al powders were mechanically synthesized for 210 minutes by mechanical alloying (MA) method. Respectively 2, 4, 6 and 8 % ratios of Al powders were synthesized in NiTi alloy powders via milling. Alloyed powders were pressed at 200°C and sintered at 1100°C for 2h. In determining and characterizing the properties of powders and sintered parts; SEM, Optical Microscope, XRD and DSC analyzes were applied, respectively. Optical Microscope images show the fine grains obtained in NiTi+Al 8% alloys. In XRD analysis B₂, B₁₉, AlNi, AlNi₂ and AlNi₂Ti peaks were seen. The XRD results show the needed 2θ peaks could be achieved with sintering of NiTi+Al alloy system. The results show that the obtained parts will be used in implant technology with their fine and pure structures.

Key words: NiTi+Al alloys, Mechanical synthesis, AlNi₂/Ti/Al3Ni/AlNi phases

1. Introduction

Shape Memory Alloys (SMA) are formed by chemical composition of approximately equal elements of Ni and Ti and named as Nitinol. These alloys can be used in industrial and medical applications due to their good damping effect, unique shape memory properties, excellent biocompatibility and abrasion resistance [1-3]. It is generally preferred in the production of NiTi alloys from SMAs due to the ease of casting. Failure
to achieve the desired mechanical properties (such as ductility, fracture resistance and superelasticity) in the samples produced by the melting process, however, results in the emergence of alternative production methods besides this production method [4]. Hydrogen, which can be formed in the structure, can directly affect the mechanical properties, especially because of NiTi alloys are directly affected by environmental conditions during their production [5]. For these reasons, the use of different production methods such as powder metallurgy (PM) is becoming increasingly widespread in order to minimize the effect of the environmental factor in production conditions [6, 7]. PM methods are suitable for use in NiTi alloys; production methods such as hot isostatic pressing, metal injection molding and spark plasma sintering are at the forefront of these fields [8, 9].

Mechanical alloying (MA) from PM application areas is the most common method. MA is used in the addition of a different alloy powder into a manufactured alloy powder. So, MA methods directly effect determination and replacement of the composition of used alloy system [10]. MA is being used because of the productivity. It provides in changing atomic ratios of the Ni and Ti chemically present in the SMA system and in the synthesis of alloy elements [11-13]. The main purpose of using this technique is to obtain a homogeneous structure and improve the material properties. Also, this technique gives ease of synthesis of the same or a different material. Unique physical, chemical and mechanical properties can be obtained in materials by means of the MA method to produce nano-sized particle production [14].

In this study; SMA NiTi powders and pure Al (2, 4, 6, 8 wt.%) powders were milled for 210 minutes by MA technique. To characterize the resulting NiTi-Al powder mixtures, OM, SEM, XRD and DSC analyzes were performed. The effects of Al addition at different ratios in prealloyed NiTi alloy were investigated after sintering at 1100°C for 2 h.

2. Materials and Methods

Prealloyed NiTi SMA powders and pure Al powders were used in the MA process. Prealloyed NiTi powders were obtained by NANOVAL firm. Table 1 shows atomic and weight percentages of NiTi powders used in the study. Table 2 shows the average particle sizes of NiTi and Al powders used in MA studies, respectively. NiTi powders have 35 μm and Al powders have less than 5 μm size distribution.

Figure 1 shows a schematic representation of NiTi+Al alloy production processes. In the first stage; prealloyed NiTi and pure Al powders were mixed (in a steel cap at 15 min) for homogenization. In the second stage; NiTi+(2, 4, 6, 8 wt.%) Al powders were mechanically alloyed for 210 min [15]. In the third stage; mechanically alloyed powders were pressed to obtain green parts. In the third stage, green parts sintered at 1100°C for 2h for obtaining the red samples.
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### Table 1

<table>
<thead>
<tr>
<th>Elements</th>
<th>Ni</th>
<th>Ti</th>
</tr>
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<tbody>
<tr>
<td>Weight%</td>
<td>54.5</td>
<td>45.5</td>
</tr>
<tr>
<td>Atomic%</td>
<td>50.6</td>
<td>49.4</td>
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### Table 2

<table>
<thead>
<tr>
<th>Powders</th>
<th>Average particle size distribution (μm)</th>
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<tbody>
<tr>
<td>NiTi</td>
<td>35</td>
</tr>
<tr>
<td>Al</td>
<td>5</td>
</tr>
</tbody>
</table>

Figure 1. Schematic representation of Pre-preparation, MA, Pressing and Sintering processes of NiTi+Al powders

Scanning Electron Microscopy (SEM), Energy Distribution Spectrometer (EDS), X-Ray Diffraction (XRD), Optical Microscope Images and Differential Scanning Calorimetry (DSC) analyzes were performed to characterize the samples. SEM images were obtained via JEOL model JSM-6060LV device. The DSC analyzes were aimed at determining the phase structure, microstructure transformation and particle morphology of the alloys by taking 5-10 mg of each sample and performing a scanning rate of 5 °C/min. In the DSC analyzes, Exstar S11 7300 model device was used liquid nitrogen (to cool the samples to -70 °C). DSC analysis were performed between -70 °C to + 350 °C temperature range. XRD analyzes were
performed by APD 2000 PRO XRD model Xray using 0.04 step and copper cathode (CuKα) at 20-90° and 2θ scan range. A LEICA reverse microscope was used to examine the images of the sintered NiTi sample. Sintering was carried out at 1100°C for 2 hours in a high purity Argon atmosphere.

3. Results and Discussion

Figure 2 shows SEM image of NiTi powders. The use of gas atomization method in the production of NiTi and Al powders ensures that the powders have a spherical structure. Powders in spherical structure are known to play an important role in facilitating the process of pressing and sintering [16, 17].

Phase transformation temperatures and hysteresis curves are important for SMA applications. In the study, DSC analysis was performed to determine the transformation temperatures of prealloyed NiTi powders (Fig. 3). In the DSC, the austenite temperature starts at about -30 °C and ends at +10 °C. This transformation represents the standard transformation for NiTi alloys [18]. According to the DSC curve, these transformation have austenite phase at room temperature (~26 °C) and have these alloys superelasticity [19]. Fig. 4 shows the XRD analysis results of sintered NiTi+Al 2, 4, 6, 8% (Fig. 4.a), prealloyed NiTi powders and mechanically alloyed NiTi+Al (2, 6%) powders (Fig. 4.a). Fig. 4b shows the prealloyed NiTi powders are formed only the peaks of the B₂ phase (2θ = 42.1° and 2θ = 43.2°). In addition, the XRD analysis results of NiTi + Al (2 and 6) powder mixtures were examined, the highest peak values were determined at 2θ = 42.1°. The reduction and expansion of peak intensities after milling support the formation of amorphous phase and the grain refinement [12]. This shows that the fine grains and amorphous phase begins to form. In Fig. 4.a seen the XRD analysis results (NiTi+Al (2, 4, 6, 8 %)) of sintered parts. In the
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XRD analysis B₂, B₁₉, AlNi, Al₃Ni and AlNi₃Ti peaks were seen (in Fig. 4.b). The XRD results show the needed 2θ peaks could be achieved with sintering of NiTi+Al alloy [20-22].

![Figure 3. DSC analysis of prealloyed NiTi powders](image)

In Fig. 5 a - d show the OM images of sintered NiTi+Al (2, 4, 6, 8%) alloy at 1100°C for 2 hours. In Fig. 5.a shows the NiTi-Al 2% alloys OM structure. In microstructure different sized grains were seen. Fig. 5.c shows the NiTi + Al 8% sintered sample OM image. In OM image, the fine grain structure have been obtained the increasing of the Al ratio, decreasing of the grain sizes. The fined grains have been obtained in NiTi+Al %8 alloys, because of the Al powders are making barrier effects to NiTi powders during sintering.
Figure 5. Optical microscope images of sintered samples, a) NiTi+Al 2%, NiTi+Al 4%, NiTi+Al 6%, NiTi+Al 8%

4. Conclusion

Results of NiTi+Al alloys, mechanically synthesized and then sintered at 1100°C for 2h could be summarized as follows;

- DSC analysis shows the austenite temperature starts at about -30°C and ends at +10°C. This transformation represents the standard transformation in NiTi alloys and gives the super elasticity property.

- The XRD analyzes of powders have only the peaks of B2 main phase (2θ = 42.1° and 2θ = 43.2°). In the powders XRD analysis results, the reduction and expansion of peak intensities after milling show the formation of amorphous phase and the grain refinement with milling.

- XRD analysis results (NiTi + Al (2, 4, 6, 8 %)) of sintered parts give the B2, B19, AlNi, Al3Ni and AlNi2Ti peaks. The XRD results show the aimed peaks achieved with sintering of NiTi+Al alloy system at 1100°C for 2h.

- It has been obtained the grain refinement with the increasing the Al ratio to sintering at 1100°C for 2 hours. The fined grains structure have been obtained in NiTi+Al 8% alloys, because Al powders are making barrier effects to NiTi powders sintering.

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References


