Heat Flow Behaviours of Distaloy AE Alloys Produced by Powder Metallurgy

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Abstract

The alloying techniques are classified with pre-alloyed, pre-mixed and diffusion-bonded powders. Distaloy AE alloy used in this study is type of diffusion-bonded alloy including of Fe, Ni and Mo alloying elements and has high strengthening microstructures. In this study; hardness, density and heat flows distaloy AE samples prepared by powder metallurgy method performed. As hardness and density measurements of these samples, Brinell hardness measurements and Archimedes water displacement technique performed and heat flows of these alloys determined using Differential Scanning Calorimetry (DSC) device. These results supported by SEM images, EDS spectrums of alloys and also, crystal structures of these alloys determined by XRD patterns.

Keywords: Distaloy AE, Powder metallurgy, Heat flow, Surface characterization

Toz Metalurjisi Metoduyla Üretilen Distaloy AE Alaşımlarının İısı Akışı Davranışları

Öz

Alaşımları teknikleri ön işlemleri, önkarışımları ve difüzyon-bağlı tozla olmak üzere sınıflandırılır. Bu çalışmada kullanılan Distaloy AE alınını Fe, Ni ve Mo alınım elementleri içeren bir difüzyon-bağlı alınım çeşididir ve yüksek dayanıklı mikroyapıya sahiptir. Bu çalışmada toz metalürjisi metoduyla üretilen Distaloy AE alınımlarının sertlik, yoğunluk ve sısl akişları çalışılmıştır. Bu malzemelerin sertlik ve yoğunluk ölçümleri Brinell sertlik ölçümleri ve Archimedes suyun kaldırma tekniğini kullanılmıştır ve Diferansiyel Taramalı Kalorimetre (DSC) cihazı kullanarak alınımların sısl akişları saptanmıştır. Sonuçlar alınımların SEM fotoğrafları, EDS spektrumlarıyla desteklenmiştir ve ayrıca alınımların kristal yapıları XRD desenleriyle saptanmıştır.

Anahtar Kelimeler: Distaloy AE, Toz metalürjisi, İısı akişi, Yüzey karekterizasyonu

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1. INTRODUCTION

Powder metallurgy method that is one of producing of iron and iron derived alloys includes of three main stages powder/powder mixture preparation, shaping of prepared powder mixture in order to obtain green bulk product and densifying of shaping of green bulk products for acquiring densely bulk products from green products. This sintered product has more densely structure compared to green bulk products [1-5].

Pre-alloyed or diffusion bonded alloys includes of Fe, Ni alloying elements and shows high strengthening structure [6-8]. Distaloy AE alloy used in this study has a diffusion bonding powder structure and on the structure of Distaloy AE alloy, between Fe and dispersed alloying element into Fe-matrix, metallurgical binding structure happens. This situation provides high strengthening to structure [8-10].

Heat flow occurs always from a region having high temperature to a region having lower temperature and happens by conduction, convection and radiation [11,12]. On the solids, heat flow comes true via conduction. In this study; heat flows and hardness of distaloy AE produced by powder metallurgy method have been investigated and relationship between heat flow and mechanical property of these alloys has been determined.

2. MATERIAL AND METHOD

In this study; Distaloy AE alloy powder that is obtained from Höganäs in Sweden utilized. The chemical composition of this powder includes of 0.01 of C%, 4.00 of Ni%, 1.50 of %Cu 0.50 of Mo% and Fe in balance.

Distaloy AE powder mixed with lubricant pressed on the 400 and 600 MPa pressures using traditional pressing technique on the 10×15×70 mm molds. Pressed these green bulk products annealed at 600 and 600 °C temperature for 30 min in order to debond of grains in the alloy and provide uniform temperature distribution according to literature [13-15]. After then, samples sintered at 1200 °C, for 2 hours under N₂ atmosphere. Heating and cooling rate selected as 5 °C/min. In this work; as hardness test. Brinell hardness measurements of these alloys performed and calculated following Equation (1).

\[
HB = \frac{2P}{\pi d} \left( d - \sqrt{d^2 - \frac{D^2}{4}} \right) \quad \text{(N/mm}^2) \quad (1)
\]

In this Equation (1) P and d are referred as ball load and ball diameter of indentator that is contacted to surface of alloys. D is referred as diameter of indentation on the surfaces of these alloys. HB gives Brinell hardness values of these alloys. Ball load and ball diameter have been given as 31.25N and 2.5 mm, respectively. Brinell hardness test performed using BMS Digirock Hardness Tester.

The density measurements of Distaloy AE samples prepared on the 400 and 600 MPa pressures performed via Archimedes water displacement technique; pure water as medium was utilized. Samples cut to small prismatic samples in order to put chamber of assay balance with with 0.0001 sensitivity and Samples firstly dry weighted on assay balance, after, weighted samples were taken in pure water and were boiled to boiling point of water (100 °C) and also waited for 4 hours at this temperature due to homogeneous dispersing of water into pores in the microstructures of samples. After then, samples cooled in water to room temperature and cooled samples dried with clear dishtowel and were individually weighted in air suspended in pure water. Density and porosity measurements of Distaloy AE alloy samples were calculated with acquiring weighting values of these samples. Equations (2-5) of density and porosity measurements of these samples were given in below,

\[
\text{Bulk Density (} \rho_{\text{bulk}} \text{)} = \frac{w_k}{w_p} \times \rho_{\text{liquid}} \quad (2)
\]

\[
\text{Apparent solid density (} \rho_{\text{apparent}} \text{)} = \frac{w_k}{w_{K-A}} \times \rho_{\text{liquid}} \quad (3)
\]

\[
\text{Porosity (} \rho_{\text{porosity}} \text{)} = \frac{w_p - w_k}{w_p} \times 100 \quad (4)
\]

\[
\text{True density (} \rho_{\text{true}} \text{)} = \frac{w_k}{w_p} \times \rho_{\text{liquid}} \quad (5)
\]
Apparent porosity \( = \frac{W_D - W_K}{W_D} \times 100 \) (4)

Water adsorption \( = \frac{W_D - W_A}{W_K} \times 100 \) (5)

From equations; \( W_K, W_A \) and \( W_D \) are defined as dried weight, suspended weight in pure water, weighted in air, respectively. Density measurements performed using Scaltec Sec31 with precision of 0.0001g.

Heat flows of sintered Distaloy AE samples and powder Distaloy AE have been investigated using DSC (Differential Scanning Calorimetry). These heat flows of alloys supported via SEM images, EDS spectrums and XRD patterns of alloys.

Figure 1. The SEM image of Distaloy AE powder (40 μm)

3. RESULTS AND DISCUSSION

For Brinell hardness values of Distaloy AE alloys, it was obtained that distaloy AE prepared on the low pressure has 106.25 N/mm\(^2\), whereas other Distaloy AE sample has 177.32 N/mm\(^2\). When pressure increased, Brinell hardness values of these samples increased. With pressure increasing, porosity closed, grain bonding occurred and Distaloy AE samples have more strengthening microstructure.

On the Table 2; bulk and apparent solid density (g/cm\(^3\)), apparent solid porosity (%) and also water adsorption (%) values of Distaloy AE alloy samples prepared on the 400 and 600 MPa pressures were given. It was observed that bulk and apparent solid density of samples increased when pressure increased. Apparent porosity and water adsorption (%) of samples decreased. With increasing of pressure; pores closed, grain bonding occurs, therefore microstructures of samples gain dense and strengthening structures. Maximum bulk and apparent solid density are obtained on the sample prepared on the high pressure (6.38 and 6.55 g/cm\(^3\), respectively) and Minimum apparent and water adsorption are recorded as 2.60 and 0.41 %, respectively.

Table 1. The density measurements of Distaloy AE samples prepared on the 400 and 600 MPa pressures

|                  | 400 MPa | 600 MPa |
|------------------|---------|---------|
| Bulk density (g/cm\(^3\)) | 6.18    | 6.38    |
| Apparent solid density (g/cm\(^3\)) | 6.41    | 6.55    |
| Apparent porosity (%) | 3.67    | 2.60    |
| Water adsorption (%) | 2.2     | 0.41    |

Figure 2. The heat flows of powder and sintered Distaloy AE alloys

In Figure 2; the heat flows of powder and sintered Distaloy AE alloys are given. The heat flows of powder and Distaloy AE prepared on the 400 MPa pressure were recorded close to each other. On the austenitic stainless steel prepared on the 600 MPa pressure; heat flow has higher values than that of Distaloy AE powder and prepared on the 400 MPa pressure. Heat values of these alloys were given according to literature [16]. In this study; for all samples prepared on the 400 and 600 MPa pressures.
samples; first, all heat flow plates were arranged at temperature of approximately 25 °C, after, at the temperature range of approximately 550-450 °C, values of all heat flow plates decreased at time range from 15 to 40 sec, after then, at medium temperature of approximately 450 °C for Distaloy AE powder and prepared on the 400 MPa pressure and at temperature range from 550 to 450 °C for distaloy AE prepared on the 600 MPa pressure; on the values of heat flows decreasing occur at time of 275 sec. On the heat flow of Distaloy AE powder; values of heat flows firstly decreases at temperature of 350 °C and then, increases at temperature of 325 °C, at temperature range from 300°C to 200°C and at time range from 1265 to 1690 sec, on the values of heat flow, frustrating occurred. For Distaloy AE alloy prepared on 400MPa pressure; at temperature range from 205 to 108 °C and at time range from approximately 990 to 1065 sec., values of heat flow of this sample, decreasing occurs. Maximum heat flow was recorded on the distaloy AE alloy prepared on the 600 MPa pressure. (-3.86279 mW at 277 sec at temperature of 57.667 °C)

In Figure 3 and Table 2, the SEM images and EDS spectrums of sintered Distaloy AE alloys are given. Surface appearances of both of distaloy AE samples have smooth. With pressure effect and heat treatment, porosity into alloy structure closes and more grain binding happens. Via heat treatment; increasing of atomic vibration causes to diffusion and more atoms acts in the contact area and hence, bonding surface happens. Occurred bonding surface extends, therefore, sintering necks occurs [17,18]. this situation observed that surface of Distaloy AE alloy prepared on the 600 MPa pressure has smoother appearance than that of other alloy sample and recommended that this situation has a role on the higher heat flows of this samples according to the other samples.

From EDS spectrums in Table 2; ratios of C, Ni, Cu alloying elements decreased, but ratios of Fe and Mo alloying elements increased. This situation is observed o the XRD patterns. Sintering at high temperature, liquid phase that is caused to densification occurs between alloying elements and carbon atoms [6,19]. More carbon atoms on the structure of alloys lead to transformation martensite structure that is Cu-rich network. In addition, Ni-rich causes to austenitic structure. Cu melts down at almost 1085 °C temperature. Diffusion of Ni atoms happens into the melting of Cu. Martensitic structure having of both of Cu and Ni rich have a significant a role on the reinforcing of alloys [6].

|                  | 400 MPa | 600 MPa |
|------------------|---------|---------|
| C                | 4.11    | 3.7     |
| Ni               | 4.21    | 4.09    |
| Cu               | 1.56    | 1.28    |
| Mo               | 0.3     | 0.34    |
| Fe               | 89.82   | 90.59   |
In Figure 4, the XRD patterns of powder and sintered Distaloy AE samples are observed. On the all XRD patterns of these alloy; it was seen that Fe and Ni alloying elements in microstructures of alloys were preponderated to Cu alloying element. XRD pattern of this alloy powder; (111) cubic crystal structure (at 43.4°) varied to (011) crystal structure on the both of sintered Distaloy AE alloys and at 50.59-51.85° range. It was also observed that (020) cubic crystal structure (Cu, Ni) on the distaloy powder was not seen on both sintered Distaloy AE alloys [20,21].

- On the SEM images of these alloys; decreasing porosity and therefore grain bonding occurs with increasing of pressures and XRD patterns of of these alloys; Fe and Ni alloying elements in the structures of samples were dominated according to Cu alloying element.

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6. REFERENCES

1. Panda, S.S., Singh, V., Upadhyaaya, A., Agrawal, D., 2006. Sintering Response of Austenitic (316L) and Ferritic (434L) Stainless Steel Consolidated in Conventional and Microwave Furnaces, Scripta Materialia, 54, 2179–2183.

2. Giménez, S., Vagnon, A., Bouvard, D., Van der Biest, O., 2006. Influence of the Green Density on the Dewaxing Behaviour of Uniaxially Pressed Powder Compacts, Materials Science and Engineering A 430, 277–284.

3. Yılmaz, R., Ekici, M.R., 2015. Üretim Parametrelerinin Düşük Alaşımlı TM Çeliklerin Sertlik ve Aşınma Özelliklerine Etkisi 3rd International Symposium on Innovative Technologies In Engineering and Science (ISITES2015) Valencia-Spain, 2545-2554.

4. Zarebski, K., Putyra, P., 2015. Iron Powder-based Graded Products Sintered by Conventional Method and by SPS, Advanced Powder Technology, 26, 401–408.

5. Al-Qureshi, H.A., Galiotto, A., Klein, A.N., 2005. On the Mechanics of Cold Die Compaction for Powder Metallurgy, Journal of Materials Processing Technology, 166, 135–143.

6. Abdoos, H., Khorsand, H., Shahani, A.R., 2009. Fatigue Behavior of Diffusion Bonded Powder Metallurgy Steel with Heterogeneous Microstructure, Materials and Design, 30, 026-1031.

7. James, W.B., OtBrien, R.C., 1986. High Performance Ferrous PM Materials: The Effect
of Alloying Method on Dynamic Properties. Progress in Powder Metallurgy. Princeton NJ: MPIF.

8. Alzati, L., Bergmark, A., Andersson, J., 2005. Fatigue Performance of PM Steel in Assintered State. Presented at PMAI Conference, Mumbai, India, 9.

9. Lindskog, P., 2013. The History of Distaloy, Powder Metallurgy, 56:5, 351-361.

10. Öksüz, K.E., Kumruoğlu, L.C., Tur, O., 2015. Effect of Sic on the Microstructure and Mechanical Properties of Sintered Distaloy DC Composites, 5th International Biennial Conference on Ultrafin Grained and Nanostructured Materials (UFGNMS15), Tehran-Iran, Procedia Materials Science 11, 49-54.

11. http://labman.phys.utk.edu/phys221core/modules/m9/regulation.html

12. https://study.com/academy/lesson/heat-flow-in-solids-fluids.html

13. Pandya, S., Ramakrishna, K.S., Annamalai A.R., Upadhyaya, A., 2012. Effect of Sintering Temperature on the Mechanical and Electrochemical Properties of Austenitic Stainless Steel, Materials Science and Engineering A, 556, 271–277.

14. Butković, S., Oruč, M., Šarić, E., Mehmedović, M., 2012. Effect of Sintering Parameters on the Density, Microstructure and Mechanical Properties of the Niobium-Modified Heat-Resistant Stainless Steel GX40CrNiSi25-20 Produced by MIM Technology, Materiali in tehnologije/Materials and Technology 46, 2, 185–190.

15. Butković, S., 2013. Sinterability And Tensile Properties of Nickel Free Austenitic Stainless Steel X15CrMnMoN 17 11 3, Technical Gazette 20, 2, 269-274.

16. Göbel, A., Vidi, S., Klinker, F., Hemberger, F., Brütting, M., Ebert, H.P., Mehling, H., 2017 Method for the Thermal Characterization of PCM Systems in the Volume Range from 100 ml to 1000 ml, International Journal of Thermophysic 38(67), 12.

17. Yao, B., Zhou, Z., Duan, L., Xiao, Z., 2016. Compressibility of 304 Stainless Steel Powder Metallurgy Materials Reinforced with 304 Short Stainless Steel Fibers, Materials, 9(161), 1-11.

18. Yıldızlı, K., 2015. Investigation on the Microstructure and Toughness Properties of Austenitic and Duplex Stainless Steels Weldments Under Cryogenic Conditions, Materials and Design, 77, 83–94.

19. Karwan-Baczewska, J., 2015. Processing and Properties of Distaloy SA Sintered Alloys with Boron and Carbon, Archives of Metallurgy and Materials, 60(1), 41-45.

20. Acar, A.N., Ekşi, A.K., Ekicibil, A., 2019. Structural and Physical Properties of Sintered Distaloy AE Alloy Compacts, UDCS’19 Fourth International Iron and Steel Symposium, 4-6 April, Karabuk, 231-233.

21. Acar, A.N., Ekşi, A.K., Ekicibil, A., 2019. Specific Heat Capacity Behaviours of P/M Distaloy AE Alloy Compacts, International Conference on Condensed Matter and Materials Science ICCMMS-2019 14-19 October 2019 Adana, 7-13.