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EFFECT OF POST-HEAT TREATMENT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Cu-Fe-Co POWDER ALLOY FABRICATED BY HOT PRESSING

In this study, the powder mixture which consists of Cu-Fe-Co was produced by using the method of hot pressing technique. In addition, effect of heat treatment process on microstructure and mechanical properties of this alloy was investigated. Following the hot pressing process applied on the samples, heat treatment was carried out at 950°C for two different dwelling times (90 and 180 minutes). Measured density values were considered as physical characteristics, while hardness and fracture strength values were considered as mechanical characteristics. It was observed that porosity rates of hot pressed samples were decreased by increasing of temperature and pressing parameters. In this study it was observed that Cu was spread in matrix and filled the micro porosities. Hardness values were determined to be decreased as a result of grain growth after heat treatment process. That 4 fold increased elongation % values which were achieved by TRS experiments were observed as well.

Keywords: Diamond cutting tools, Cu-Fe-Co alloy, Powder metallurgy, Hot pressing, Heat treatment

1. Introduction

Thanks to the cutting properties, particular reinforced metal matrix composites are widely used. Because of their good mechanical properties such as, toughness, a hardness and strength, ceramic particles are preferred as reinforcing materials for the composites [1-9]. Diamond cutting tools are widely used to cut and process natural stones and they called as metal matrix composites. These tools are consisting of diamond which performs cutting and a metal matrix which holds and supports diamond particles during tool life and they are produced by powder metallurgy techniques [10].

For a high performance diamond cutting tool, matrix must support the diamond continuously and firmly during cutting and bonding between interface of matrix and diamond must be very strong [11]. In these days, pre-alloyed powders are intensively used as bonding matrix in the production of diamond cutting tools [12,13]. Thanks to pre-alloying process that mixes powders more homogenously, undesired segregation for tool is prevented and therefore tool properties are improved. In addition, activation energy can be reduced during atomic diffusion by using pre-alloyed powders [14,15].

Grain growth can be described as growing of the grains by diffusion occurring in time within the material, as a result of annealing of material at a temperature higher than its crystallization temperature. Porosities in the microstructure of PM products are reduced as a result of grain growth which occurs during sintering process and higher density is achieved [16]. Therefore grain growth is a desired case at specific rates [17].

Copper is widely used in powder metallurgy production because of its advantages such as raising the conductivity, improving the properties of alloys, its accessibility and being cheap [18,19]. In addition, copper helps increasing the density and toughness by filling the pores in the alloy with its lower activation energy [20], therefore contributes the providing integrity of matrix [21]. Ni is used in PM alloys in order to reduce the temperature of sintering process [22]. Also Ni improves the mechanical properties of matrix at diamond cutting tools [23]. Co is used in diamond cutting tools because of its features like workable with diamond at high temperatures, perfect diamond retention and compatible wearing with diamond. Nevertheless, because of its price instabilities and its hard attainability, researches on the materials which could replace the Co were carried out [24]. Especially Fe-Cu-Co alloys have been developed in order to reduce the amount of Co [25,28]. MX1480 pre-alloyed powder which was developed as an alternative for Co in diamond tool production is widely used by diamond cutting tool industry.

Interface bond between diamond and matrix is one of the key factors that determine the tool life. Therefore, in this study we purposed to improve the bond structure between matrix and diamond and also to reduce micro porosities by grain growth which is occurred as a result of heat treatment that is applied to matrix alloy 3% Ni+20% Cu+MX1480 which were produced by hot pressing method.

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2. Experimental studies

2.1. Material and method

In the starting matrix powders, Ni with an average particle size 2µm, Cu with an average particle size 63 µm and as alternative of Co, commercially available MX1480 powder which consist of Next100 (50% Cu-25% Fe-25% Co) + 20% Fe with an average particle size 3 µm were used. Powders that were used in the compound are summarized in the Table 1. By weight 3% Ni+20% Cu+MX1480 compound used as the matrix in produced segments that was sintered by hot pressing process. Content of matrix and its theoretical density was given in Table 2. The theoretical density was calculated the rule of mixture.

TABLE 1

Properties of Powders

Powder	Average Particle Size	Code	Company
Ni	2 µm	Ni 2800 A	Eurotungstene, France
Cu	63 µm	Cu 1197	GGP Metal Powder, Germany
MX1480	3 µm	MX1480	Eurotungstene, France

TABLE 2

Chemical compositions of alloy

	Ni	Cu	MX 1480	Theoretical Density [g/cm^3]
Wt%	3	20	77	8,43

Powder mixtures were weighed by using precision scales and mixing process was applied for 30 minutes by using a turbula mixer (Celmak Group 7T, Turkey). PEG 400 (Polyethylene Glycol) at a rate of 1,5wt% was added in the powder mixture in order to reduce friction forces during hot pressing and to provide a homogenized mixture. Previously, powders were weighed 20 grams, and then green segments were produced with a pressure of 300 MPa by using double-effect hydraulic press (Dim-Net WP-45SA, Korea). After that, green segments were placed within graphite dies and hot pressed under vacuum atmosphere by a PLC controlled direct hot pressing machine (Zhengzhou Golden Highway, SMVB 80, China). Following that production process was completed by applying a heat treatment to the samples in a furnace (Prothem, PLF 120/27, Turkey) under argon atmosphere at 950°C for 90 minutes and 180 minutes. All samples were left to free cooling after heat treatment. Sample codes and parameters of production were given in the Table 3.

2.2. Characterization

The fabricated samples were characterized for hardness, density, TRS, SEM and XRD. Surface of the samples were polished with 600 mesh wet sandpaper by using grinding device (Metkon Forcimat, Turkey). Density measurements were determined according to the standard of ASTM B311-92 Archimedes principle. Hardness of the samples were measured in Brinell hardness terms (HB10) by using a 2,5 mm diameter tip with 62,5 kg of load (EmcoTest, Durajet, Austria). Surface of the samples were etched by a special etcher which consists of 30% HNO_3 + 30% acetic acid + 6.5% H_3PO_4 + 33.5% H_2O after polishing and then microstructure investigations were performed. TRS tests were performed according the standard of ASTM B528-83a by a test machine (Shimadzu, AG-IS 100 Kn, Japan) with Trapezium X software. SEM (JEOL JSM-7001F, Japan) was used for investigations of microstructures of samples and fracture surfaces and for analysis of EDS. XRD analysis were performed by using X-ray device (Rigaku RadB-DMAX II, USA) for analyzing the phases in the microstructure.

TABLE 3

Production Parameters

	Hot Pressing Parameters			Post-Heat Treatment Parameters	
Sample	Pressure [MPa]	Temperature [°C]	Time [min.]	Temperature [°C]	Dwell Time [min.]
A1	20	700	4		
A2	20	700	4	950	90
A3	20	700	4	950	180
B1	30	800	4		
B2	30	800	4	950	90
B3	30	800	4	950	180

3. Results and discussion

3.1. Microstructure

Microstructure images of hot pressed and post-heat treated samples are shown in Fig. 1 (A group) and Fig. 2 (B group) was taken with a high and low magnification. When the SEM images of A and B samples are studied, it was seen that porosity amount was decreased by increasing of sintering temperature and pressure [29]. Porosities have formed at macro size around Cu particles. It was also seen that micro porosities have formed in the MX1480 because of its smaller particle size. It was also seen that porosities have formed at macro size around the Cu particles in the B1 sample too; however formation of micro porosities in the MX1480 is less than A1. Therewithal, Cu particles are spread in the matrix easily and amount of porosity were decreased thanks to higher hot pressing parameters of sample B1 [18,19].

Analysis of SEM images of the heat treated A2 and B2 samples at 950°C for 90 minutes was shown that grain growth was occurred in the microstructure. When the high magnification SEM image of A2 (Fig. 1b,e) was investigated, it was seen that micro porosities between the MX1480 particles have transformed into macro porosities by changing of shape and Cu started spreading in the matrix. More grain growth has occurred in A2 microstructure compared to the B2 sample because of its

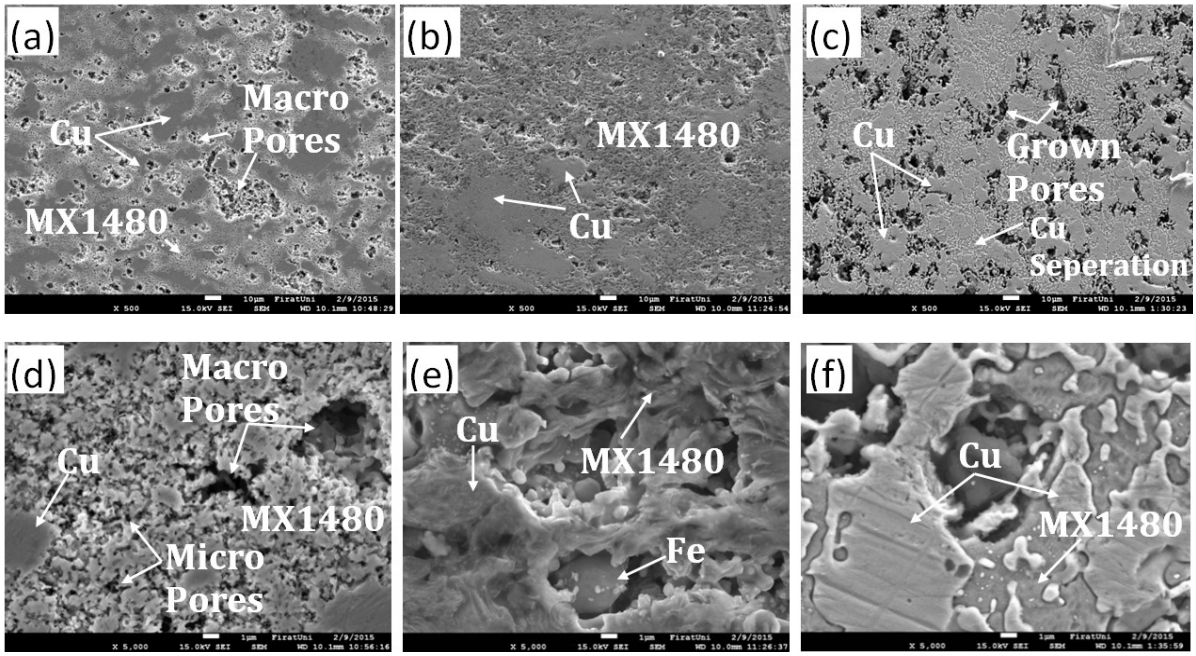

Fig. 1. SEM images of A samples that taken low a) A1, b) A2, c) A3 and high magnification d) A1, e) A2, f) A3

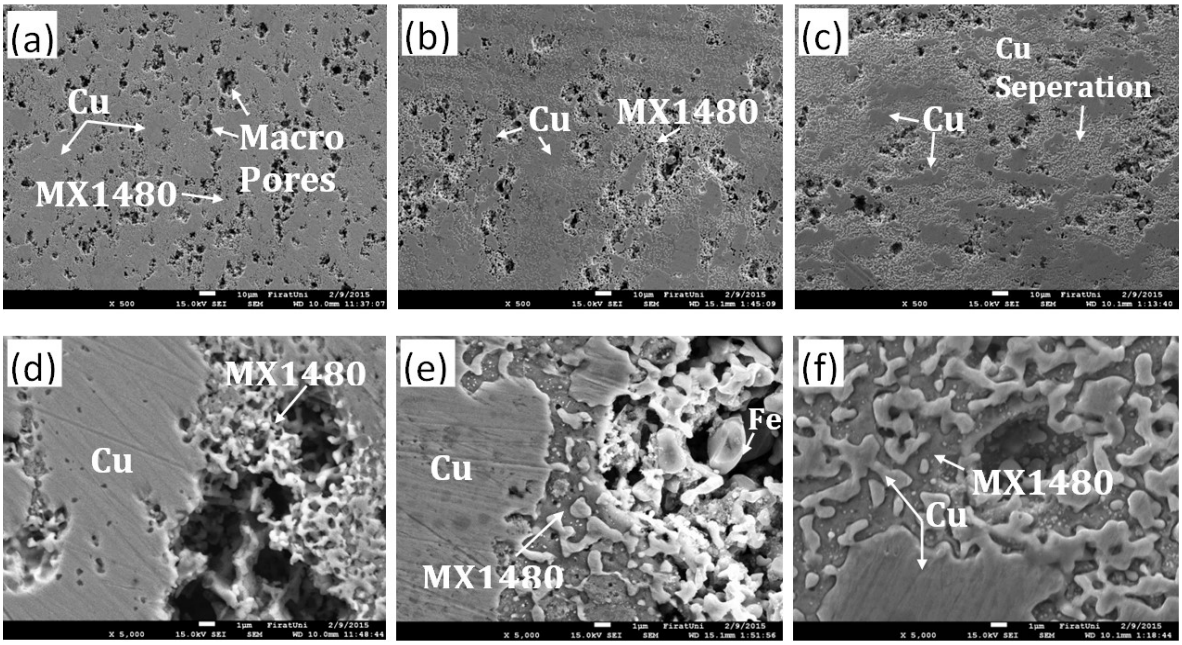

Fig. 2. SEM images of B samples that taken low a) B1, b) B2, c) B3 and high magnification d) B1, e) B2, f) B3

prior lower micro porosity content. It was seen that, during grain growth the size of macro porosities which were existed before heat treatment have increased. The amount of macro porosity has increased significantly because of the grain growth in the microstructure of A3 (Fig. 1c,f) sample which has been heat treated at 950°C for 180 minutes. Moreover, grain growth had continued and copper had spread greatly in the microstructure of A3 sample. It was seen that, compared to A2 sample, amount of micro porosity was decreased substantially and size of macro porosities were increased in the microstructure of A3. Spreading of Cu in the matrix microstructure B3 (Fig. 2c,f) is more than matrix microstructure of A3.

3.2. Density and hardness

Results of the density and hardness measurements of the samples were given in the Table 4. From the obtained results, it was seen that hardness and density values of A1 and B1 samples were increased by increasing the hot pressing temperature and pressure. Density value of A1 was measured to be 8,06 g/cm^3 whilst this value was measured to be 8,31 g/cm^3 for B1. Density and hardness values of A2 and B2 were decreased because of the grain growth and increased amount of porosity which were formed after the heat treatment. Same situation is valid for A3 and B3 too. This situation can be clearly seen in the SEM im-

ages Fig. 1 and Fig. 2. The heat treatment which made after hot pressing, grown the pores and increased the porosity rate.

TABLE 4

Density, Hardness and Porosity Rates

Sample	Hardness [HB10]	Density [g/cm^3]	Porosity Rate [%]
A1	161	8,06	4,39
A2	113	7,82	7,24
A3	106	7,85	6,89
B1	185	8,31	1,43
B2	121	8,15	3,34
B3	101	8,15	3,34

Hardness value of A1 was measured to be 165 HB10 and hardness of B1 was measured to be 185 HB10. It was observed that heat treatment has caused a decrease in the hardness and in the density values of the samples. After heat treatment for A2 and A3 samples density values were measured to be 113 HB10 and 106 HB10 respectively. Density values of B2 and B3 samples were measured 121 HB10 and 101 HB10.

3.3. Fractured surfaces

Fractured surface images and elemental analyses of A and B samples are given in the Fig. 3 (A samples) and Fig. 4 (B samples). It was understood from fractured surface images that how hot pressing parameters affects the fracturing features of the samples.

From the fractured surface image of A1 given in Fig. 3a,b macro and micro porosities can be clearly seen. Neck formation between Cu particles and porosities are seen in the images clearly. It was also understood that, fracture has occurred between MX1480 particles. Height difference between fractured surfaces can clearly be seen from the images too. These observations prove that, the fracture pattern is a brittle fracture. Fracture surface image of B1 that is given in Fig. 4a,b shows the effect of high temperature and pressure parameters on the samples. Height difference between fractured surfaces are reduced compared to sample A1 thanks to decreased porosity amount and increased bonding between particles.

When the fractured surfaces of A2 which is shown in Fig. 3c,d and B2 that is shown in Fig. 4c,d are studied, it was

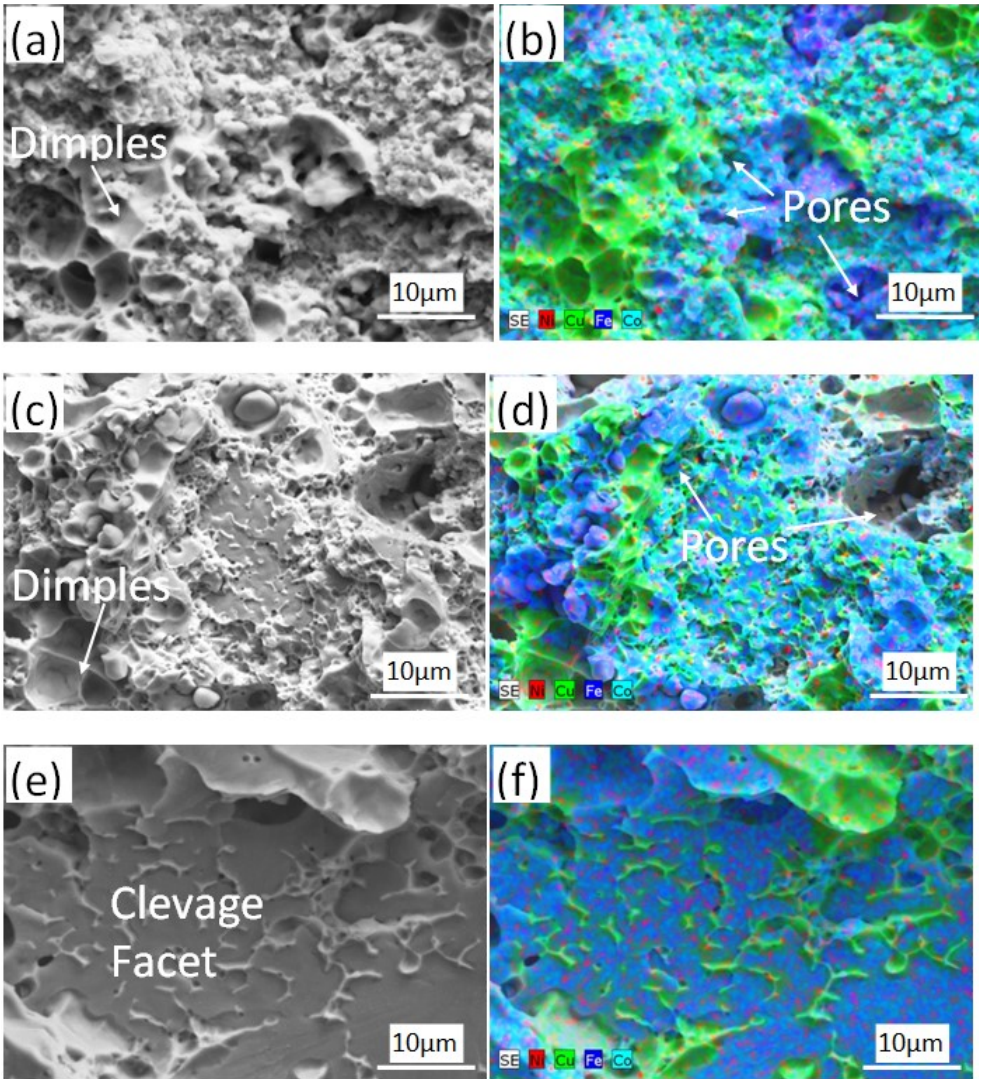

Fig. 3. SEM images and MAP analysis of fractured surfaces A samples a-b) A1, c-d) A2, e-f) A3

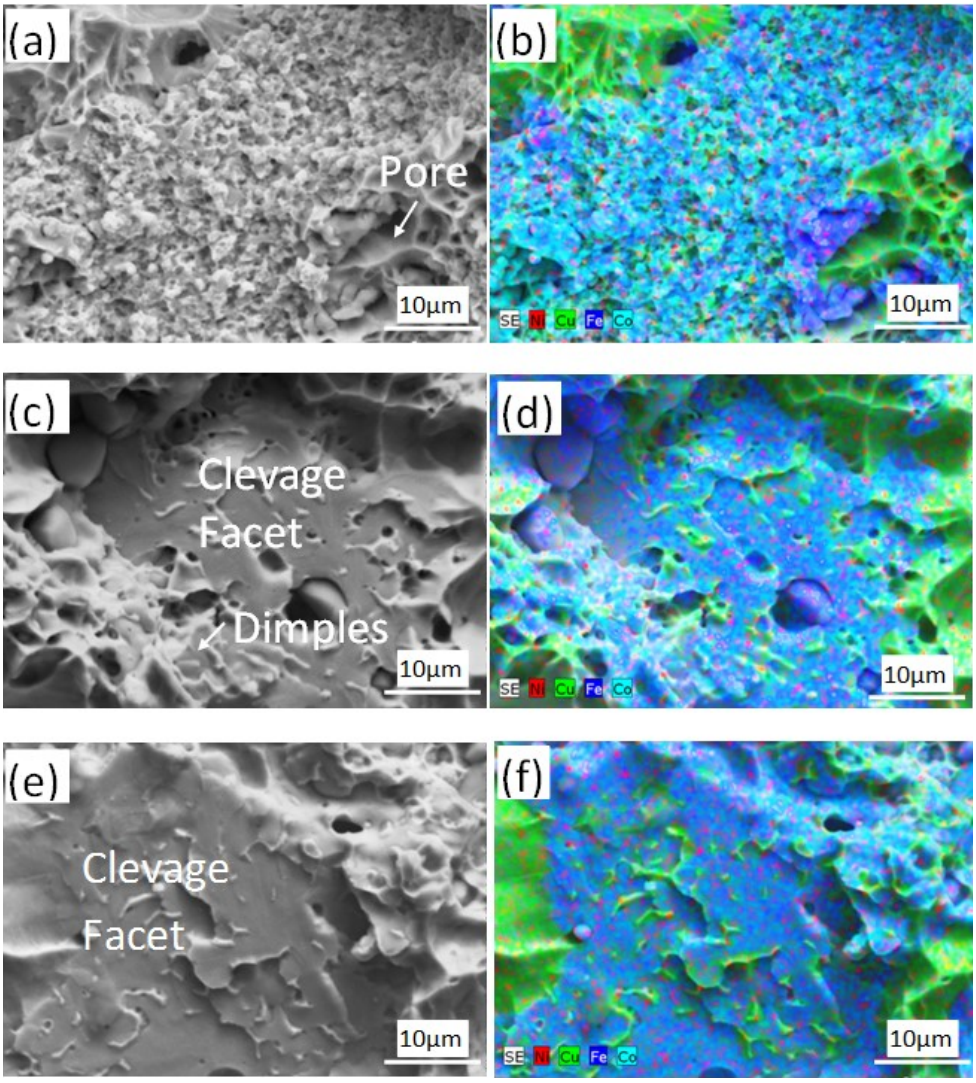

Fig. 4. SEM images and MAP analysis of B samples a-b) B1, c-d) B2, e-f) B3

observed that bonding between particles and neck formation between Cu particles have increased because of grain growth. Also it was observed that local cleavage surfaces have formed as a result of a decrease in porosity amount. It was seen that cleavage surfaces have larger area because of increasing bonding between powder particles. Therewithal, Fe particles in the MX1480 powder result in the formation of transient areas.

Fractured surface images of A3 shown in Fig. 3e,f and B3 shown in Fig. 4e,f reveals that fracture characteristic has completely changed because of the grain growth and spread of Cu in the matrix as a result of heat treatment which was applied at 950°C for 180 minutes. A planar cleavage surface has formed because of micro porosities filled by Cu particles. Also, it was seen that internal fracture axes have formed in the cleavage surfaces of B3 sample.

3.4. TRS results

TRS results of samples are given in the Fig. 5. As it can be concluded from the graphs, bending strength of samples have increased by increasing the hot pressing temperature and the pressure. Strength of A1 and B1 samples were measured consecutively 1050 MPa and 1380 MPa, elongation % of samples were measured 2,5 and 3,8. Higher strength values were obtained by increased the bonding strength between particles as a result of decreasing amount of porosity. This situation can be seen in the microstructure images Fig. 1 and Fig. 2. Furthermore, as it can be seen in the strength-elongation graphs, brittle fracture has formed in A1 sample whilst more ductile fracture has formed as a result of a plastic deformation in B1 sample compared to A1. Elongation of heat treated A2, A3 and B2, B3 samples have significantly increased as a result of grain growth and spread of Cu in the structure compared to A1 and B1 samples which were not heat treated. Elongation % values were measured 12% for A2, 11,5% for A3, 13,1% for B2 and 11,9% for B3 sample. A slight decrease has occurred strength values of samples which have heat treated for 180 minutes. Especially decreasing strength values of B samples are higher than decreasing strength values of A samples. This is because of more amount of grain growth in B samples compared to A samples. Hardness measurements support this situation too. Plastic deformation which was occurred in

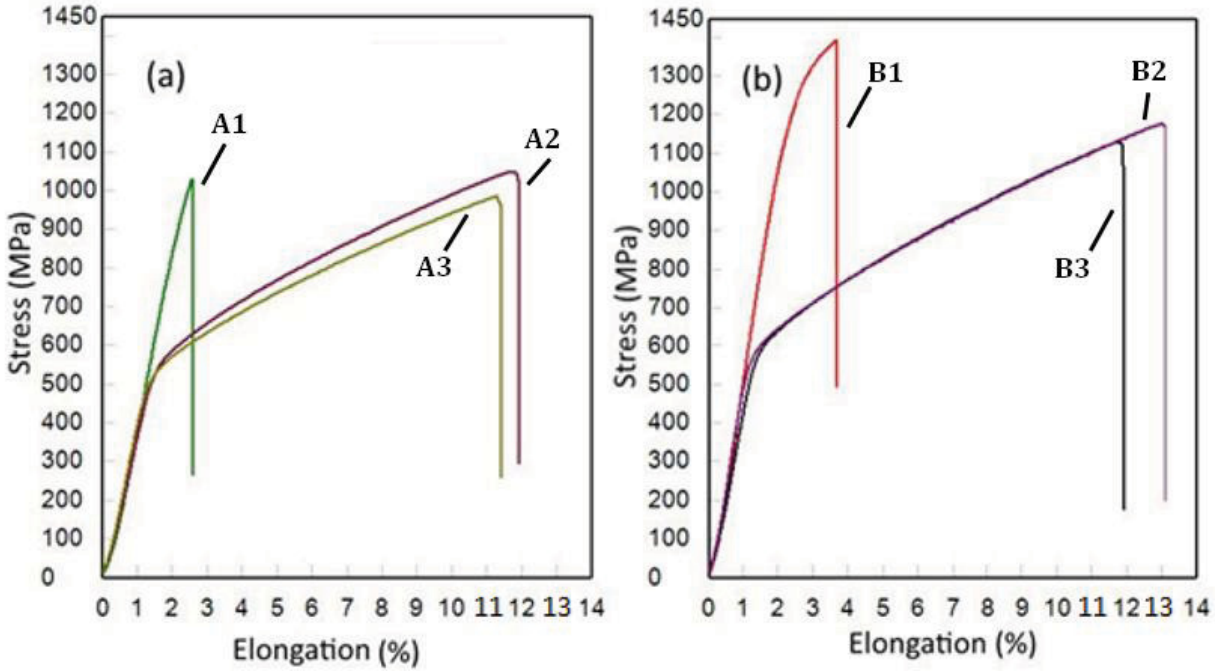

Fig. 5. Strength-Elongation graphs of produced segments a) A samples b) B samples

the microstructure of heat treated samples is seen clearly in the Strength-Elongation graphs. Furthermore, it was concluded that the heat treatment has caused lower strength values by creating a notch effect as a result of increased porosity amount.

3.5. XRD Results

XRD results of the hot pressed and then heat treated samples are given in the Fig. 6. Phase analysis reveals that Co_3Fe_7 intermetallic phase and $FeCu_4$, Fe_3Ni_2 solid solutions have formed in the micro structure. [30]. Pure Fe and Cu were seen in the microstructure as well. It was observed that amount of $FeCu_4$ solid solution in the microstructure has increased by increasing the hot pressed temperature and pressure. The results of the hardness, density, TRS and XRD measurements considered together, it was understood that this phase has decreased the density and hardness of the samples but nonetheless has increased the ductile of the matrix.

4. Conclusions

In this study, produced matrix which consist of 20% Cu, 77% MX1480 (25% Fe+50% Cu-25% Ni -25% Fe) and 3% Ni by weight was produced successfully via hot pressing process and

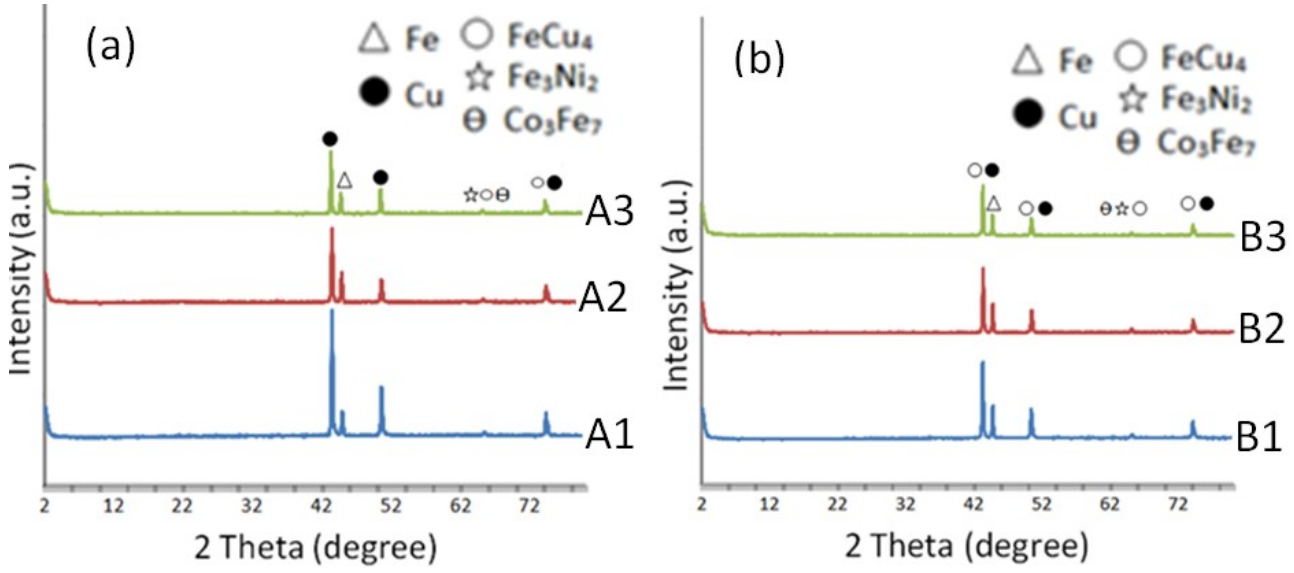

Fig. 6. XRD patterns of a) A samples b) B samples

then for two different dwell time a heat treatment was applied to samples. Effects of applied heat treatment on the samples have investigated, therefore results that listed below were revealed:

- Hardness, density and bending strength values of samples have increased by increasing hot pressing temperature and pressure
- Amount of porosity has decreased as a result of increased hot pressing temperature and pressure
- As a result of the heat treatment which was applied 950°C for 180 minutes, distortion of Cu particles in the matrix has significantly raised and this rise has increased the elongation of samples distinctly while it has decreased hardness values of samples
- According to the results revealed by experiments, without changing chemical compound of matrix mechanical properties have been improved and elongation values of samples have increased as a result of applied heat treatments at 950°C for 90 minutes and 180 minutes
- As a result of TRS experiments, thanks to decreasing micro porosity rate the bonding between matrix and diamond improved by applied heat treatments

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