Sintering Temperature and Deposition Orientation Effects on Mechanical, Physical Properties and Geometric Distortion of Cu–Ni Single and Multi Material Indirect Sintering Products

Susilo Adi Widyanto^{*)} and Eko Sutarto

Department of Mechanical Engineering, Faculty of Engineering, Universitas Diponegoro, Semarang 50275, Indonesia

^{*)}E-mail: susilo70@yahoo.com

Abstract

Development of multi material mechanical parts is constantly undertaken to increase functional aspectsas well as life cycle. One example is the use of bimaterial which is widely used as a temperature contactor. This paper presents mechanical, physical properties and geometric distortion of Cu-Ni indirect sintering products used to develop Cu-Ni bimaterial products. The experiment was executed with the following method: firstly, Cu and/or Ni powders were deposited into cast iron powder as the supporting powder. Secondly, it was heated in a furnace with varying temperatures of 870 °C, 900 °C and 930 °C with a holding time of four hours. Lastly, deposition orientation was varied to observe the effect on the occurence of shrinkage. To initiate the multi materials sintering process, single material sintering of Cu and Ni powders was conducted. The experiment results showed that the geometric distortion of the sintering products was influenced by deposition orientation. The Cu and Ni products shrinkage were 49% and 35.33%, respectively. Although the melting temperature of Cu and Ni is close, the binding mechanism of the sintered product did not occur. The significant difference of shrinkage levels was the main factor for the binding mechanism failure between Cu and Ni materials.

Abstrak

Pengaruh Temperatur Sintering dan Orientasi Deposisi pada Sifat Mekanis, Fisik dan Distorsi Geometri Produk Sintering Tidak Langsung Single dan Multi Material CU-Ni. Pengembangan produk-produk mekanis multi material senantiasa dilakukan untuk meningkatkan aspek fungsional dan umur. Salah satu contoh adalah produk bimaterial yang secara luas digunakan sebagai kontaktor temperatur. Paper ini memaparkan sifat mekanis, fisik dan distorsi geometri produk sintering tidak langsung berbahan Cu-Ni yang digunakan untuk pengembangan produk bimaterial. Eksperimen dilakukan dengan metode sebagai berikut: pertama, serbuk Cu dan atau Ni dideposisikan ke dalam serbuk besi cor sebagai serbuk penyangga. Kedua, serbuk terdeposisi dipanaskan di dalam furnace dengan variasi temperatur 870 °C, 900 °C dan 930 °C dengan waktu penahanan selama empat jam. Tahap akhir, orientasi deposisi divariasikan untuk mengamati pengaruhnya pada penyusutan yang terjadi. Untuk mengawali proses sintering multi material, sintering material tunggal dilakukan untuk mengamati sifat fisik dan mekanisnya. Mengacu pada penelitian sebelumnya, sintering multi material antara serbuk Cu dan Ni dilaksanakan. Hasil eksperimen menunjukkan bahwa distorsi geometri pada produk sintering dipengaruhi oleh orientasi deposisi. Penyusutan produk Cu dan Ni masing-masing adalah 49% dan 35,33%. Meskipun temperatur leleh Cu dan Ni berdekatan, mekanisme ikatan produk sinter tidak terjadi. Perbedaan penyusutan yang signifikan merupakan faktor utama kegagalan dalam pembentukan ikatan antara material Cu dan Ni.

Keywords: Cu, indirect sintering, multi materials, Ni

1. Introduction

In application, Cu-Ni alloys are widely used for marine applications due to their excellent resistance to seawater corrosion, high inherent resistance to bio-fouling and good fabrication ability [1]. While, infield instruments and controls, Cu-Ni has long been used as a thermal bimaterial actuator.

Because of its extensive applicability its mechanical and thermal properties should be further investigated. The strengthening mechanisms in bimetallic Cu/Ni thin layers were investigated by Shehadeh et al. [2]. They used a hybrid approach that links the parametric dislocation dynamics method with ab initio calculations. The hybrid approach is an extension of the Peierls-Nabarro (PN) model on bimaterials, where the dislocation spreading over the interface is explicitly accounted for. The Cu/Ni bimaterial system is modelled as two semi-infinite homogenous and isotropic regions connected at the interface. As a result, they reproduced several MD simulation trends and made further predictions about the strength of Cu/Ni laminates, without reliance on empirical potentials. In experiment, mechanical properties and thermal stability of electrodeposited Cu-Ni multilayers were studied by Tokarz et al. Their work found that the multilayered Cu/Ni coatings exhibit greater hardness than single Ni and Cu layers when the thickness of the bi-layers is approximately a few nanometers. The maximum hardness values were measured when the bi-layer thickness was around 10 nm and was about 25% greater than the Ni single layer hardness [3].

The idea of multi material product development instantly drives the creation of new manufacturing processes such as layer manufacturing. Development of a multi material selective laser sintering process was performed by Beaman *et al.* [4]. This product is called as *functional graded materials*, FGMs. For instance, FGMs technology is applicable to mining tools which comprises of two materials to yield toughness and high wear resistance without a welding process.

According to Beaman *et al.* [4], the development of a multi material layer manufacturing process was determined by the capability of modifying product powder deposition. Various powder deposition methods have been developed for layer manufacturing application. Vibration, gravitation and pressure assisted flow are single material powder deposition methods based on hopper nozzle construction [5-6]. The use of hopper nozzle as a powder deposition sub-system changed the procedures in layer manufacturing processing. Powder deposition must be performed in a series of steps depending on how many product materials are used. In every layer, the deposition procedure was initiated by depositing product powder and finished by depositing supporting powder.

In the sintering process, the use of multi materials product powder has consequences, for instance, how to produce a binding mechanism among the different materials with a specific melting point. Multi materials sintering of Cu-Ni based on single material analysis was studied by Shimosaka *et al.* [7]. They reported that the same thermal absorption coefficient of the materials was the key to the occurrence of a bonding mechanism between Cu and Ni particles.

During the sintering process, the powder experiences the alterations of free energy, shrinkage and mass transport phenomena. The level of shrinkage is determined by the initial green part density. Low green part density yields greater shrinkage than that of the sintered product with the higher green part density [8]. Compaction is the process whereby the initial density of the green part is increased. In multi material sintering, the shrinkage depends upon the thickness layer of each green part [9]. Furthermore, shrinkage is the consequence of microstructure transformation as well as transport mechanism [10].

Total free energy of the system also decreases proportionally to the particles total area [11]. Capillarity forces are the driving forces of solidification and surface tension, while gravitation drives shape distortion [12].

The research was an initial step to develop the smart fan blade which used Cu-Ni materials produced by an indirect sintering process (Figure 3). This method represented multimaterial deposition indirect sintering (MMDIs), a kind of layer manufacturing, developed in previous research. This paper presents the mechanical and physical properties and also the geometric distortion of single and multi materials of Cu-Ni products.

2. Methods

In this research, Cu and Ni powders were used as product material, while cast iron powder was employed as the supporting powder. Chemical compositions of Cu and Ni powders are presented in Table 1 [13].

In the deposition process, the particle shape of powder determines the actual density of the green part which in turn influences the sintered product density [5]. In this research, the particle shapes of the powders were observed using optical microscopy. The results of particle shape observation is shown in Figure 1.

Specimens manually fabricated using the indirect sintering process represents the multi material layer manufacturing process. For single material products, Cu or Ni powders was deposited into aluminum molding (dimensions illustrated in Figure 2) which was placed around cast iron powder as the supporting powder in a metal can. The surface of the deposited powder was swept and then the molding was removed after the supporting powder was deposited around the molding. Procedures of the fabricated specimen are schematically illustrated in Figure 3. The sintering process was performed with varying temperatures of 870 °C, 900 °C and 930 °C and deposition orientation. The particle sizes

Content	Composition
	(%)
Cu Powder, particle size of $<63 \ \mu m$	
comlexometric	99.7
insoluble in nitric acid	0.02
Р	0.001
Ag	0.002
As	0.0005
Fe	0.005
Mn	0.001
Pb	0.01
Sb	0.001
Sn	0.01
Ni Powder, particle size of 10 μ m	
Ni	>99.5
С	< 0.28
Fe	< 0.002
Со	< 0.001
Si	< 0.001
Cu	< 0.001
Mg	< 0.001
As	< 0.001
S	< 0.001
Zn	< 0.001
Р	< 0.001
Cd	< 0.0003
Bi	< 0.0003
Mn	< 0.001
Sn	< 0.0003
Pb	< 0.0003
Sb	< 0.0003
Са	< 0.005

 Table 1. Chemical Compositions of Cu and Ni Powders [13]

of the supporting powder also varied at 75-104 μm (150 mesh) and 105-149 μm (100 mesh).

After sintering, the specimens were cleaned from the adhering supporting powder and the dimensional was measured. The dimensional difference between the product of molding and the actual sintered product shows the shrinkage phenomenon. By measuring the weight, the bulk density was then calculated. To observe the mechanical strength, the sintered specimens was tested using the tensile strength test according to ASTM D638 standard (Figure 4).



Figure 2. Specimen Dimensions



Figure 3. Procedures of Specimen Fabrication Using Indirect Sintering Process



Figure 4. Tensile Strength Test Specimen based on ASTM D638



Figure 1. The Particle Shapes of a) Cu Powder, b) Ni Powder, c) Cast Iron Powder

doi: 10.7454/mss.v16i2.1407/Makara J. Sci. 16/2 (2012) 122-128

3. Results and Discussion

Single material sintering products. Sintering energy (sintering temperature and holding time) directly affects the final density of sintered products. During sintering, the green part experiences shrinkage (reduction of volume) caused by decreased inter particle porosity as a result of holding time. In correlation to stress concentration, due to the presence of pores, the level of density is proportional to mechanical properties such as tensile strength. The experiment results which represent the correlation betweeen tensile strength and sintering temperature is illustrated in Figure 5.

Figure 5 shows that the tensile strength of the Ni product is higher than the Cu product. The tensile strength closely correlated to material density. By using remaining pieces of tensile strength specimens, the result of the density testing is depicted in Figure 6. This figure shows that the density of the Ni product is higher than that of the Cu product. During sintering, the flow-ability of particles increases in time which is the consequence of phase transformation. Meanwhile, gravitation becomes the driving force of flow direction. Because of the higher bulk density, the effect of the driving force on the Ni product was higher than the Cu product.

Initial density of the green part greatly affected the final density and shrinkage of the sintered product. Because of the un-compaction process, the initial density of indirect sintering green parts were only influenced by deposition parameters such as particle shape, particle size and bulk density of material. Observation of the particle shapes showed that the Cu particle was more irregular than the Ni particle (Figure 1). This was the reason why the initial density of Ni powder was higher than Cu density.

After being sintered, the density of Cu and Ni as the function of sintering temperature, in which the density of the Ni product was higher than the density of the Cu product (Figure 6). The initial density of the green part



Figure 5. The Tensile Strength of Cu and Ni Products Produced by Varying the Sintering Temperature

also affected the occurrence of shrinkage. The experiment results showed that shrinkage of the Cu product was higher than the Ni product at 49% and 35.33%, respectively.

Maximum shrinkage occurred in the vertical direction for Cu as well as Ni products. Not only shrinkage but the sintered product also experienced geometric distortion. In orientation as depicted in Figure 7a, the product experienced maximum shrinkage in the vertical direction and geometric distortion in the lateral direction (Figure 7b). Results of measuring the temperature distribution of the building part (the wall as the heat generator) showed that temperature accumulation occurred in the center radial location of the building part.

Temperature was also a function of the vertical position: the higher the position the lower the temperature. Due to the difference of temperature distribution that was not uniform, the product also experienced non-uniformed shrinkage.

Green part orientation also affected the shrinkage and geometric distortion. As illustrated in Figure 8a, product experienced the thickness reduction (vertical orientation) and geometric distortion which shapes facing upwards. If the sintering process is executed in a



Figure 6. Correlation between Product Density of Cu- Ni and Sintering Temperature (Holding Time of 4 Hours, Particle Size of Supporting Powder 150 Micron, Particle Size of Product Powder 100 Micron)



Figure 7. (a) Green Part Orientation, (b) Cu Product Experiences Shrinkage in Vertical Direction and Geometric Distortion in Lateral Direction

furnace, temperature accumulates in the center of the building part in radial as well as vertical position. In the formation of powder deposition illustrated in Figure 8a, the upper product surface received higher temperature than the bottom (powder was deposited in the lower mid vertical position of the building part). This implied that shrinkage increased as the function of vertical position in which the upper surface experienced maximum shrinkage (Figure 8b).

The effects of sintering temperature on the occurence of shrinkage and geometric distortion are depicted in Figure 9. This figure shows that geometric distortion is proportionally increased by sintering temperature.

Besides the physical and thermal properties, the differences of shrinkage and geometric distortion of Cu-Ni was also affected by the initial density of the green part. Initial density of Ni powder was higher than Cu powder, which consequently presented different particle shapes. Figure 10 shows the comparison of Ni and Cu sintered products. The geometric distortion of the Cu sintered product was higher than the Ni sintered product. It was caused dominantly by the non uniformity of shrinkage.

The result of micrography observation showed that the Cu product of the indirect sintering process was more porous than the Cu product produced by the rolling process (Figure 11). This condition was caused by the uncompaction process which minimized the inter particle contact forces. In the indirect sintering process, gravitation is a driving force which stimulates inter particle bonding mechanism.

In the indirect sintering process, deposited product powder was sintered around the supporting powder, so that the characteristics of the supporting powder



Figure 8. Cu Product Experienced Shrinkage and Geometric Distortion



Figure 9. Geometric Distortion of the Sintering Product Fabricated at Varying Sintering Temperatures for 4 Hours: a) 870 °C, b) 900 °C, c) 930 °C

influenced the temperature distribution in building part. The effect of the supporting powder particle size of cast iron powder (sintering temperature of 900 °C, holding time of 4 hours) on the microstructure of Cu product is illustrated in Figure 12. The particle size of the supporting powder correlates directly to the thermal conductivity of the powder. The smaller the particle size the greater the thermal conductivity [14].

The net of sintering energy affects the inter particle bonding mechanism which determines mechanical characteristics. The effect of the sintering temperature on the microstructure of Cu products is shown in Figure 13.

Multi materials of Cu-Ni products. The experiment results of the Cu-Ni sintering product showed that the role of the driving force of the sintering process determines the particle formation bonding mechanism. The horizontal parallel formation of materials (Figure 14) failed to produce the bonding mechanism. This was indicated by the relative movement between materials in the boundary area as a consequence of different shrinkages.



Figure 10. a) Ni Sintered Product, b) Cu Sintered Product



Figure 11. Micrograph Observation of Cu Material Fabricated by Colling Process (Magnification of 200x)



Figure 12. The effect of Supporting Powder Particle Size on the Microstructure of Cu Product: (a) 75-105 Micron, (b) 105-150 Micron (Magnification of 200x)

In the vertical formation (Cu powder at the bottomillustrated in Figure 15a), Cu and Ni materials produced the bonding mechanism. These conditions indicated that the weight aspect of Ni powder was a driving force to form a bonding mechanism among particles in the boundary area.

The condition of Ni-Cu boundary materials was also influenced by the sintering temperature. At the sintering temperature of 870 °C, a crack occurred in the boundary area of Cu and Ni materials (Figure 15b). The crack can



Figure 13. The Effect of Sintering Temperature on the Micro Condition of Cu Product: a) 870 °C, b) 900 °C, c) 930 °C (Particle Size of Supporting Powder 75-105 Micron, Holding Time of 4 Hours, Magnification of 200x)



Figure 14. (a) Sintering Formation in a Building Part,
(b) Relative Movement Due to Shrinkage of Ni and Cu which Failed to Establish a Bonding Mechanism, (c) Top View of Multi Materials Sintered Product, (d) Side View of Sintered Product



Figure 15. Ni-Cu Sintered Product Produced by Varying Sintering Temperature for 4 Hours: (a) Sintering Formation in Building Part, b) 870 °C, (c) 900 °C, (d) 930 °C

be minimized by increasing the sintering temperature (Figure 15c). At sintering temperatures of 930 °C, cracks were eliminated from the boundary area (Figure 15d). These conditions indicated that the sintering temperature determines the levels of the dominant aspects between shrinkage and gravitation as the driving force of the formation of a bonding mechanism. At the sintering temperatures of 870 °C and 900 °C the bonding mechanisms of the similar particles was larger than that of the dissimilar materials so that the force of shrinkage was more dominant than the gravitation.

4. Conclusions

This work finds that the tensile strength of Ni products is higher than that of Cu products. Up to the sintering temperature of 930 °C, the stensile strength of Cu and Ni products proportionally increases with the sintering temperature. An increase of tensile strength dominantly affects increasing product density. The shrinkage of the Cu product was greater than the Ni product, at 49% and 35.33%, respectively. In multi material sintering with Cu-Ni, the shrinkage difference was the significant problem in the formation of the bonding mechanism.

Acknowledgement

This research was supported by Hibah Pascasarjana under contract number 0596/023-04.2-16/13/2012. The authors would like to thank Vighormes and Subroto for their help in preparing the experiments, as well as Syaiful and Sri Nugroho for the discussions and assistance in conducting the experiment.

References

- L. Berthagen, The Use of Cathodic Protection for Copper Alloys in Seawater Cooling Systems, Copper Development Association Inc, *http://www.copper.org*, 2012.
- [2] M.A. Shehadeh, G. Luy, S. Banerjeez, N. Kioussisy, N. Ghoniem, Philos. Mag. Lett., LXXXVII/10 (2007) 1513.
- [3] A. Tokarz, T. Fraczek, Z. Balaga, Z. Nitkiewicz, Rev. Adv. Mater. Sci. XV (2007) 247.
- [4] J. Beaman, D. Bourell, B. Jackson, L. Jepson, D. McAdams, J. Perez, K. Wood, Proceedings of the 2000 NSF Design and Manufacturing Grantees Conference, Vancouver, Canada, 2000.
- [5] P. Kumar, J.K. Santosa, E. Beck, S. Das, Rapid Prototyping J. X/1 (2004) 14.
- [6] S. Khalil, J. Nam, W. Sun, Rapid Prototyping J., XI/1 (2005) 9.

- [7] A. Shimosaka, Y. Ueda, Y. Shirakawa, J. Hidaka, KONA. XXI (2003) 219.
- [8] S. Hambir, J.P. Jog, Bull. Mater. Sci. XXIII/3 (2000) 221.
- [9] T.J. Garino, D.J. Zschiesche, M.L. Howard, Experimental Studies to Support Multi Material Sintering Modeling, Sintering 2003, Penn State University, Pennsylvania, 2003, p.864.
- [10] D. Gouvea, R.H.R. Castro, Appl. Surf. Sci. 217 (2003) 194.
- [11] S.J. Schneider, Ceramics and Glasses, Engineering Materials Handbook, ASM International, vol. 4, Ohio, 1991, p.1217.
- [12] J. Liu, R.M. German, Metall. Mater. Trans, 30A (1999) 3211.
- [13] Anon, Cu-Ni Chemical Composition Data, MERCK products, http://www.merckmillipore.com, 2012.
- [14] S.A. Widyanto, Syaiful, J. Teknik Mesin, XIII/1 (2011) 37 (in Indonesia).