

MICROSTRUCTURAL EVOLUTION OF SINTER-FORGED Fe-Cr-Mo-C ALLOY DEPENDING ON Cu ADDITION

Pre-alloyed Astaloy CrL™ (Fe-1.5 wt% Cr-0.2 wt% Mo), a commercial Fe-based alloy powder for high strength powder metallurgy products, was sintered and hot forged with additions of 0.5 wt% C and 0~2 wt% Cu. To investigate the influence of various Cu contents, the microstructural evolution was characterized using density measurements, scanning electron microscope (SEM) and electron backscatter diffraction (EBSD). Transverse rupture strength (TRS) was measured for each composition and processing stage. The correlation between Cu additions and properties of sinter-forged Fe-Cr-Mo-C alloy was discussed in detail.

Keywords: Astaloy CrL™, Fe-based alloy, pre-alloyed powder, powder forging, microstructure

1. Introduction

Powder metallurgy (PM) technique possesses a capability to manufacture near net shapes without further shaping processes such as cutting or machining. This capability gives rise to the principal advantages of reduction in the number of processing steps, in the amount of material waste, and in the manufacturing costs [1-3]. In the PM processes, pre-alloying of powders is one of important methods that provides the final PM products with greater performance [4-8]. The Astaloy CrL™ (Fe-1.5 wt% Cr-0.2 wt% Mo) used in the present study is a very popular pre-alloyed low-alloy powder especially for high strength PM products [9-11].

In the present research, C and Cu are added into the CrL alloy to improve mechanical properties. The C is an essential alloying element for many Fe-based PM alloys. Particularly, when C is added to the Fe-Cr-Mo PM alloys, such as CrL or CrM, hard chromium carbides are formed in the matrix improving its strength. In this reason, various research has been performed on CrL-C alloys [12-16]. However, CrL-C alloy has two major issues. One is the lower compactibility due to its complex and irregular shape and high hardness because it is pre-alloyed. Another issue is the decrease in density after sintering process. The maximum volume expansion of CrL is greater than that of pure Fe which produces many pores resulting in reduction of density after sintering. In order to resolve these issues, Cu element is also added to the CrL in addition to C in the present study. Cu is generally forms a liquid phase during sintering which helps densification [17]. In the present research, 0.5 wt% C

and 0~2 wt% Cu are added to the CrL, and their sintered and forged properties are characterized depending on different Cu addition.

2. Experimental

Pre-alloyed Astaloy CrL™ (Fe-1.5 wt% Cr-0.2 wt% Mo), supplied by Höganäs AB, Sweden, was mixed with 0.5 wt% C and various amount of Cu (0, 1, and 2 wt%) using 3D Tubular mixer for 30 min. The particle sizes of CrL, C, and Cu powders were <150 μm , 5 μm , and <38 μm , respectively. As a solid lubricant, 1 wt% of Zn stearate was mixed together to enhance the compactibility and to reduce the friction between powders and die. The powder mixture was compacted at 600 MPa at room temperature. The powder compacts were de-lubricated at 760°C for 30 min and subsequently sintered at 1120°C for 30 min in 80% N₂-20% H₂ atmosphere with sintering rate of 10°C/min. Then the sintered samples were cooled in the furnace to room temperature.

For the forging process, the powder mixture was pre-compacted at 400 MPa using a conventional connecting type die. The de-lubrication and sintering processes were performed in the same conditions as above, then immediately hot forged at 500~700 MPa to achieve the final density about 7.75 g/cm³.

The standard transverse rupture strength (TRS) test was performed using a specimen dimension of 31.7 mm×12.7 mm×5 mm. The microstructure was investigated using field emission scanning electron microscope (FE-SEM, Hitachi SU-70) and electron backscatter diffraction (EBSD, EDAX Hikari).

* AJOU UNIVERSITY, DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING AND DEPARTMENT OF ENERGY SYSTEMS RESEARCH, 206 WORLDCUP-RO, SUWON, GYEONGGI, 16499, KOREA

** SEOHAN ENGINEERING RESEARCH INSTITUTE, HWASEONG, KOREA

Corresponding author: byungmin@ajou.ac.kr

3. Results and discussion

Figure 1 shows density variation of both green compacts and sintered alloys depending on the amount of Cu addition. The CrL-0.5C alloy without Cu addition shows lowest value in both green and sintered density. As mentioned above, the compactibility of CrL powder is generally lower than that of Fe and Fe-based mixed powders because CrL is pre-alloyed powder with irregular and hard surface. Therefore, it can be said that as the portion of pre-alloyed powder increases, the compactibility becomes inferior.

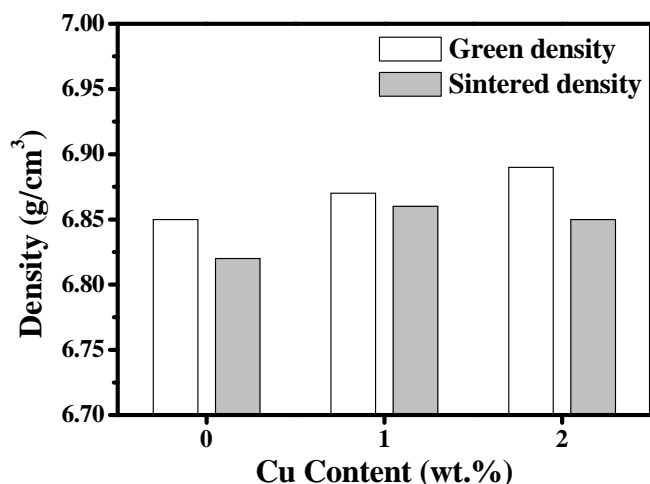


Fig. 1. Density variation of CrL-0.5C alloy depending on Cu content

As Cu content increases, the green density also slightly increases. This is attributed to the size and formability of the additive Cu element. The size and yield strength of pure Cu used in this study are about 5 μm and 70~100 MPa, respectively. When compacted at 600 MPa, the smaller Cu powders easily move to the spaces between the CrL powders, and are plastically deformed filling the interstices, resulting in the slight increase of green density with Cu addition.

In general, the density of Fe-based PM alloy increases after sintering, however in the present study, the density was decreased in all compositions compared with the green density. This density reduction is associated with Cr element in CrL powder. When 1~8 wt% of Cr is added to Fe alloy, the austenite phase exhibiting much greater thermal expansion than the ferrite phase is formed faster than ones without Cr element [18]. The excessive thermal volume expansion does not sufficiently shrink after sintering, resulting in the reduction of sintered density in the present study.

The variation of sintered density depending on Cu addition was different from the green density. When 1 wt% Cu was added to CrL-0.5C alloy, the density significantly increases. However, when 2 wt% Cu was added, the density fell slightly even though it was higher than CrL-0.5C alloy without Cu addition. Cu melts at 1094°C which is below the present sintering temperature, 1120°C. Therefore, during the sintering, Cu rapidly infiltrates pores in the compact from where it diffuses relatively easily into the CrL powder particles. In terms of the sintered density, it can be concluded that a proper addition of Cu helps to increase the

density. When an appropriate amount of Cu is added, the liquid Cu phase not only fills the pores but also acts as a diffusion path of elements, consequently resulting in enhancement of sintered density. However, when Cu is added more than the proper amount, it does not fully diffuse within the CrL in conventional sintering times and temperatures. Also, since the maximum solubility of Cu in ferrite phase is very low as 2.2 wt%, undissolved Cu causes volume expansion reducing the density.

Figure 2 shows the TRS test results of both as-sintered and as-forged specimens. The variation of as-sintered TRS exhibits similar trend as that of the sintered density. In both sintered density and TRS, CrL-0.5C with 1 wt% Cu addition showed the highest value among all compositions. With 1 wt% Cu addition, the sintered density increased by 0.4 g/cm³ which is about only 0.7% increment in the relative density. However, as-sintered TRS increased 5% with 1 wt% Cu addition. A previous study about Fe PM alloys revealed that mechanical properties were increased 1.5~2% at every density increase of 0.5 g/cm³ in the range of 6.8~7.0 g/cm³ [19]. Comparing with this previous work, the present result shows 3 times greater enhancement of mechanical property. This is attributed to the influence of Cu addition. TRS test is frequently conducted for powder metallurgy materials, and the strength value is generally affected by the presence of porosity. When 1 wt% of Cu was added in the present study, liquid phase sintering by Cu element reduced the small amount of pores, resulting in improvement of mechanical properties. Another strengthening mechanism by Cu addition is the solid solution strengthening. During sintering process, Cu is solubilized by ferrite phase which suppresses the movement of dislocations and induces solid solution strengthening.

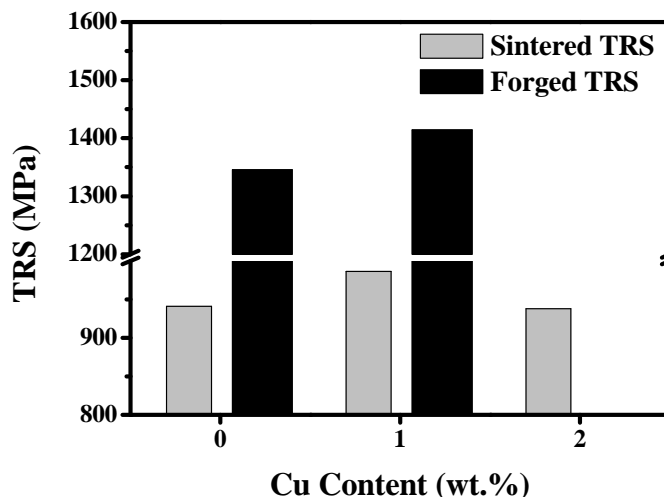


Fig. 2. Transverse rupture strength of as-sintered and as-forged CrL-0.5C alloy depending on Cu content

However, when 2 wt% Cu is added, the sintered TRS value was lowest among all compositions. This is associated with the residual Cu which decreased the mechanical property. It is further discussed in detail with SEM analysis below.

Since the CrL-0.5C with 2 wt% Cu alloy exhibited inferior properties in both density and TRS, the forging process was

performed only for two compositions, CrL-0.5C and CrL-0.5C with 1 wt% Cu. After forging, the TRS increases about 40% in both CrL-0.5C and CrL-0.5C-1Cu compared with their sintered TRS. Because the forging was performed to achieve a target density of 7.75 g/cm^3 which is near the theoretical density, it is expected that most porosities were eliminated improving mechanical properties. In addition, the materials underwent considerable amount of plastic deformation accompanied with grain refinement. This also affected significant improvement in mechanical property.

To investigate the effect of Cu addition in the CrL-0.5C alloy, the SEM microstructure was compared in all compositions as shown in Fig. 3. The CrL-0.5C contains relatively large pores with spheroid shape, while the CrL-0.5C-1Cu shows mostly narrow pores distributed along particle boundaries. The difference of pore shapes in these two materials is attributed to the distinct sintering mechanisms. The CrL-0.5C induces only

solid phase sintering, and the pores are formed at the corners of tetrakaidecahedron particles resulting in the formation of spheroid-shaped pores [20]. On the other hand, the CrL-0.5C-1Cu undergoes liquid phase sintering because of the presence of Cu, and the liquid phase Cu flows along particle boundaries. Then the Cu is dissolved into the CrL matrix so that the pores are formed where the liquid phase Cu existed along particle boundaries. When 2 wt% Cu was added, excessive amount of Cu remained even after sintering and was solidified in the inter-particle areas, resulting in decrease of sintered property as discussed in Fig. 1 and 2.

Figure 4 shows EBSD analysis results of as-sintered and as-forged materials in the compositions of CrL-0.5C and CrL-0.5C-1Cu. The average grain size of sintered CrL-0.5C and CrL-0.5C-1Cu measure from the EBSD inverse pole figure maps is $23.2 \mu\text{m}$ and $21.6 \mu\text{m}$, respectively, as shown in Fig. 4(a) and 4(b). After forging, the grain size of both CrL-0.5C and

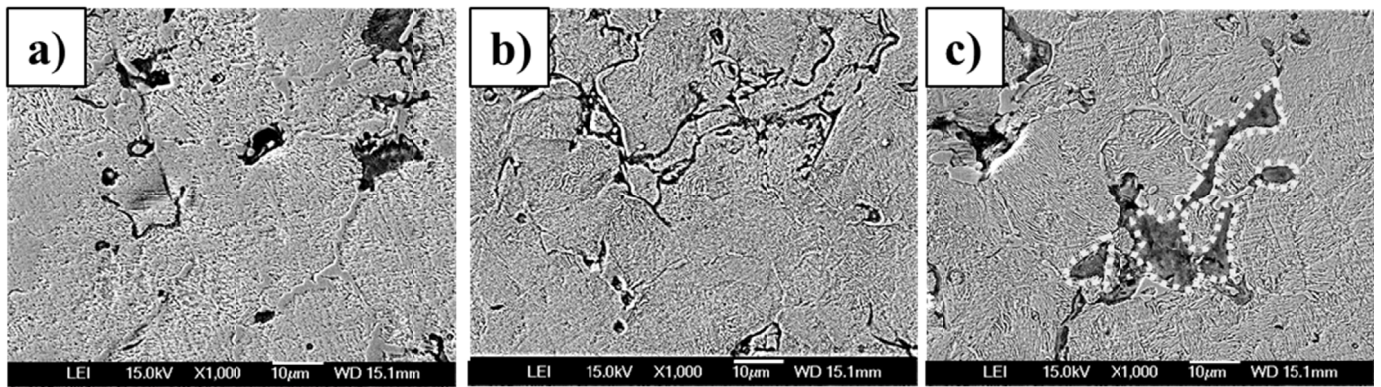


Fig. 3. SEM micrographs of as-sintered alloys showing porosity and residual Cu: (a) CrL-0.5C, (b) CrL-0.5C-1Cu, and (c) CrL-0.5C-2Cu. The dot line shows residual Cu from liquid phase

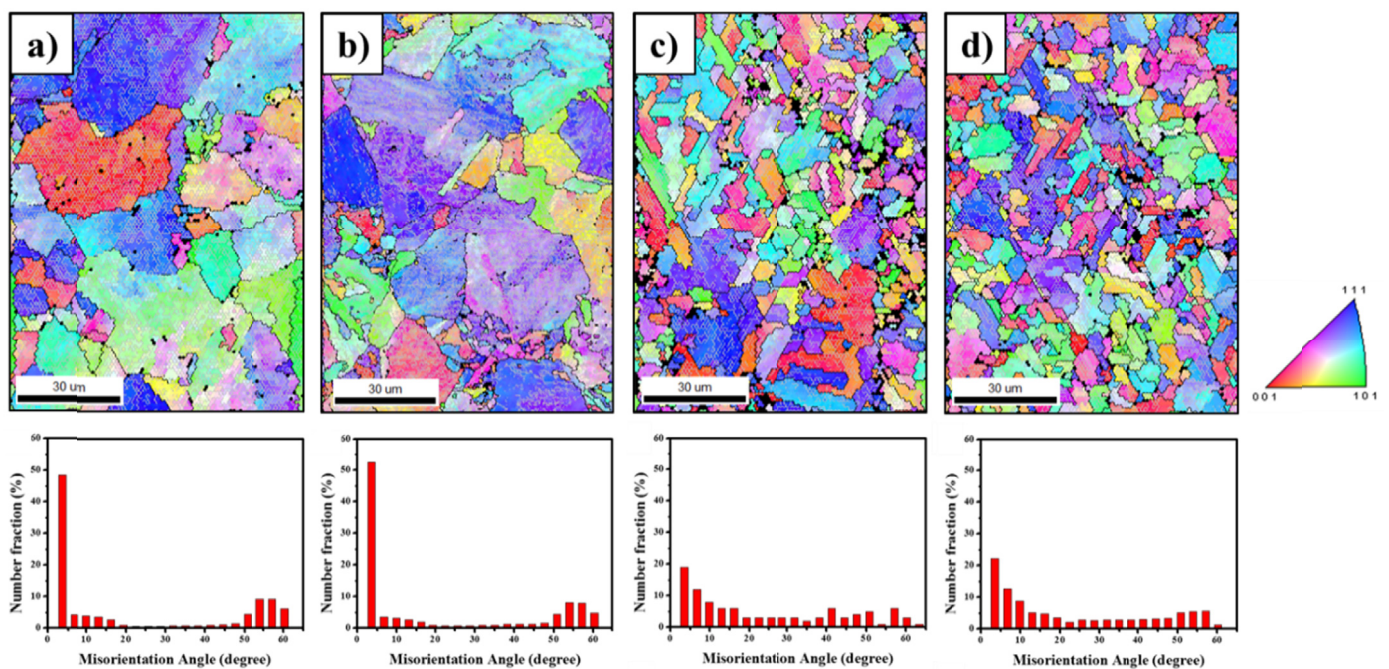


Fig. 4. EBSD inverse pole figure maps of (a) as-sintered CrL-0.5C, (b) as-sintered CrL-0.5C-1Cu, (c) as-forged CrL-0.5C, and (d) as-forged CrL-0.5C-1Cu

CrL-0.5C-1Cu was refined to 7~8 μm due to the large amount of plastic deformation during forging as shown in Fig. 4(c) and 4(d), respectively. The histograms of misorientation angle show that more than 50% of grain structure in both as-sintered materials was low-angle grain boundaries (LAGBs) while the grain structure of both as-forged materials grain structure was developed mainly into high-angle grain boundaries (HAGBs). At the beginning stage of forging, randomly distributed dislocations get arranged in a certain direction to lower the energy level. Then sets of dislocations form sub-grains, and these low-angle grain boundaries subsequently develop into randomly oriented high-angle grain boundaries as deformation progresses during forging. As a consequence of these processes, grain structure is refined after forging, and therefore the mechanical property is accordingly enhanced.

4. Conclusions

When 1 wt% Cu is added to the CrL-0.5C alloy, the mechanical property was significantly improved. During sintering process, Cu becomes liquid phase and moved to the particle boundaries. The Cu is also diffused and dissolved into ferrite resulting in enhanced sintered properties. However, when 2 wt% Cu is added, excessive Cu-rich phase segregates near particle boundaries leading to deterioration of properties. During forging process, the mechanical property increased by 40% compared with as-sintered conditions, which is attributed not only to additional densification but also to large amount of plastic deformation accompanied with grain refinement by forging. Therefore, with the addition of proper amount of Cu, enhanced property of the Fe-based commercial PM alloy (Astaloy CrLTM) was successfully achieved.

Acknowledgments

This research was supported by Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education (NRF-2018R1D1A1B07044481).

REFERENCES

- [1] W.Y. Jung, J.U. Ok, D.K. Park, I.S. Ahn, J. Korean Powder Metall. Inst. **24** 400 (2017).
- [2] N. Bekoz, E. Oktay, Mater. Des. **53**, 482 (2014).
- [3] Z.Y. Xiao, M.Y. Ke, L. Fang, M. Shao, Y.Y. Li, J. Mater. Process. Technol. **209**, 4527 (2009).
- [4] S. Nag, P. Sardar, A. Jain, A. Himanshu, D.K. Mondal, Mater. Sci. Eng. A **597**, 253 (2014).
- [5] M.A. Erden, S. Gündüz, M. Türkmen, H. Karabulut, Mater. Sci. Eng. A **588**, 201 (2013).
- [6] S. Trivedi, Y. Mehta, K. Chandra, P.S. Mishra, J. Mater. Process. Technol. **210**, 85 (2010).
- [7] D. Shanmugasundaram, R. Chandramouli, Mater. Des. **30**, 3444 (2009).
- [8] M. Teimouri, M. Ahmadi, N. Pirayesh, M. Aliofkhaezrai, M.M. Khoei, H. Khorsand, S. Mirzamohammadi, J. Alloys Compd. **477**, 591 (2009).
- [9] M. Hebda, S. Gadek, K. Miernik, J. Kazior, Adv. Powder Technol. **25**, 543 (2014).
- [10] H. Khorsand, M. Ghaffari, E. Ganjeh, Mater. Des. **55**, 979 (2014).
- [11] M. Campos, D. Sanchez, J.M. Torralba, J. Mater. Process. Technol. **143-144**, 464 (2003).
- [12] C.M. Lin, H.H. Lai, J.C. Kuom W. Wu, Mater. Charact. **62**, 1124 (2011).
- [13] G. Azimi, M. Shamanian, J. Alloys Compd. **505**, 589 (2010).
- [14] X. Zhi, J. Xing, H. Fu, B. Xiao, Mater. Lett. **62**, 857 (2008).
- [15] X. Qi, Z. Jia, Q. Yang, Y. Yang, Surf. Coat. Technol. **205**, 5510 (2011).
- [16] T. Wen, X. Hu, Y. Song, D. Yan, L. Rong, Mater. Sci. Eng. A, **588**, 201 (2013).
- [17] U. Cavdar, B. S. Unlu, E. Atýk, MTAEC9, **48**, 977 (2014).
- [18] T.P.C. Klaver, R. Drautz, M.W. Finnis, Physical Review B, **74**, 094435 (2016).
- [19] M. Sulowski, A. Cias, T. Piecozonka, Arch. Metall. Mater. **59**, 575 (2014).
- [20] M. Filgueira, D.G. Pinatti, J.N.F. de Holanda, U.U. Gomes, Mat.-wiss. u. Werkstofftech **40**, 784 (2009).