Improvement in Microstructure Homogeneity of Sintered Compacts through Powder Treatments and Alloy Designs

K. S. Hwang[a], M. W. Wu[b], F. C. Yen[c], and C. C. Sun[d]

Department of Materials Science and Engineering, National Taiwan University
1, Roosevelt Road, Sec. 4, Taipei, 106, Taiwan, R.O.C.
[a]kshwang@ccms.ntu.edu.tw, [b]r91527045@ntu.edu.tw, [c]kilua91@yahoo.com.tw,
[d]b91507037@ntu.edu.tw

Abstract

Homogeneous microstructures of the PM compacts are difficult to attain when mixed elemental powders are used. This study examined the microstructures of pressed-and-sintered and MIM products that contain Ni and Mo.Ni-rich areas, which were lean in carbon and were soft and were found easily in regular specimens. Gaps or cracks near the Ni-rich or Mo-rich areas were also frequently observed. This problem worsened when Ni and Mo particles were large and were irregular in shape. By using ball milling treatment and ferroalloy powders, the microstructure homogeneity and mechanical properties were improved. The addition of 0.5wt%Cr further improved the distribution of Ni because Cr reduced the repulsion effect between nickel and carbon. With the elimination of Ni-rich areas, more bainites and martensites were formed and mechanical properties were significantly improved.

Keywords: microstructure homogeneity, ball milling, alloy design, diffusion, chromium

1. Introduction

Nickel and molybdenum are the two elements most widely used in powder metal (PM) and metal injection molded (MIM) products. Although nickel has a high hardenability and is beneficial when added to the iron compacts, it is homogenized very little after sintering due to its slow diffusion rate in iron [1]. It has been reported that the uniformity of Ni can be improved by selecting smaller and more spherical Ni powders or using Ni-coated iron powders [2,3]. However, complete homogenization is still not achieved.

In addition to the homogenization problem of Ni, this laboratory also frequently finds Mo-rich areas on the fracture surfaces of tensile bars. This seems to be in conflict with the fact that the diffusion rate of Mo in Fe is relatively fast compared to those of nickel and other major alloying elements. Thus, there are still some questions that are unanswered, and a lot of room for improvement remains for the current PM and MIM alloy systems that contain Ni and Mo. This paper presents some of the attempts made in this laboratory on improving the microstructure homogenization and mechanical properties of PM alloys by ball milling elemental powder additives and by introducing Cr in the form of prealloyed powders.

2. Experimental and Results

To investigate the effect of ball milling Mo and Ni powders, fine carbonyl iron powder was used as the base powder. The standard MIM process was employed to prepare the specimens. To investigate the effect of Cr addition on improving the distribution of Ni, conventional pressed-and-sintered compacts were prepared using coarse diffusion alloyed Fe-4Ni-1.5Cu-0.5Mo (MPIF FD-0405) powders. The characteristics of these powders are summarized in Table 1.

To improve the mixing of Mo and Ni powder with iron powders and thus the final alloy homogenization, both powders were ball-milled for 4 hours in a Turbula mixer together with 304 stainless steel balls in the weight ratio of 1:2. The MIM feedstock was prepared and injection molded into tensile bars as per MPIF standard No. 10. Specimens were then solvent debound in heptane, followed by thermal debinding and sintering.

To investigate the effect of Cr on the Ni distribution, to the diffusion alloyed Fe-4Ni-1.5Cu-0.5Mo powders were added 0.6% graphite powder and prealloyed powders that contained 18%Cr. Tensile bars were compacted at a pressure of 500MPa to attain a green density of about 6.85g/cm^3. The specimens were debound at 550℃ for 15 minutes and then sintered at 1250℃ for 1 hour. The microstructures of all the MIM and PM specimens were examined using a scanning electron microscope (SEM) and an optical microscope. The homogeneities of Mo and Ni were inspected using an electron probe microanalyzer (EPMA).

Table 1. The characteristics of the powders used in this study

Type	Diffusion Alloyed Powder	Fe	Ni	Mo
Designation	Distaloy 4800A (Fe-4Ni-1.5 Cu-0.5Mo)	1641	Ni-123	OMP 901
Particle Size [μm]	D_{10}=16.27 D_{50}=46.85 D_{90}=100.2	D_{10}=1.95 D_{50}=4.18 D_{90}=8.48	D_{10}=2.35 D_{50}=3.69 D_{90}=6.88	D_{10}=1.23 D_{50}=2.67 D_{90}=5.88
Supplier	Hoeganaes	ISP	INCO	HCST

Effect of Size and Shape of Ni and Mo

The first attempt to improve the uniformity of Mo in the iron matrix was by ball milling the as-received Mo powder. The sharp corners of the Mo powders were slightly rounded after ball milling. This morphology change increased the tap density from 3.31g/cm^3 to 3.95g/cm^3. These changes helped lower the feedstock viscosity and facilitate the molding. The dot mapping of Mo, as shown in Figure 1, also indicated that the ball-milled Mo was more uniformly distributed in the Fe-0.5%Mo specimen, which was sintered at 1080℃ for 30 minutes. When a high sintering temperature such as 1200℃ was used, the Mo distribution was quite uniform in all specimens.

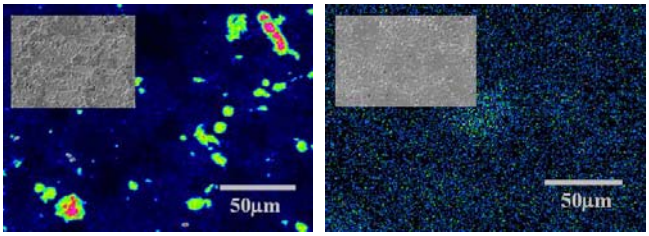

Fig. 1. The dot mappings of Mo in sintered specimens showing that ball milling improves the Mo distribution: (left) without ball milling, (right) with ball milling.

Despite the fact that the ball-milled Mo seemed to be quite uniformly distributed in sintered compacts under an optical microscope, it was easy to find Mo-rich areas at the fracture surfaces of tensile bars because these areas served as crack initiation sites. To resolve this problem, a Fe-60%Mo master alloy powder was used for Mo addition. The tensile strength attained was about 10% higher, and no Mo aggregates were found at fracture surfaces.

Effect of Cr Addition

When Ni and Mo are carefully prepared as described above, their distributions in the matrix of MIM specimens can be quite uniform. But, in the press-and-sinter compacts, the homogeneity of Ni is still poor due to the large particle size of the iron powder used. After sintering at 1120℃ for 30 minutes, Ni-rich areas were present in the pore-rich regions. These areas contained a high Ni content, between 11 and 23wt%, as was measured by the EPMA. In contrast, the center of the iron powder consisted mainly of pearlites and contained only about 0.1wt% Ni. Moreover, the EPMA also indicated qualitatively that the Ni-rich region was lean in carbon compared to that in the pearlite. This was mainly due to the high interaction energy between Ni and C [4,5]. Due to the low carbon content, the Ni-rich/C-lean areas were soft, with a hardness of only about HV 260. When 0.5%Cr was introduced, the amount of soft Ni-rich areas was reduced significantly. More martensites and bainites were formed. As a result, the tensile strength increased significantly, from 626 MPa to 801 MPa.

3. Summary

The distribution of Ni and Mo in PM and MIM alloys had not been satisfactory when regular sintering practices were employed. The ball milling treatment on elemental Mo and Ni powders helped remove the sharp corners and improved mixing. Moreover, Mo aggregates were frequently found at the fracture surfaces. This problem was resolved when Fe-Mo prealloyed powder was used. The Ni-rich areas were also observed frequently, particularly in pressed-and-sintered compacts, due to the coarse iron powder used. With ball-milled Ni powders, which helped mixing, the problem remained. It was found that adding a small amount of Cr improved the uniformity of Ni significantly and eliminated the soft Ni-rich areas. With a more uniform microstructure that consisted mainly of martensites and bainites, the mechanical properties were significantly improved.

4. References

1. K. S. Hwang, C. H. Hsieh, and G. J. Shu: Powder Metall., **45[2]**, p.160, (2002).
2. K.S. Hwang and M.Y. Shiau: Metall. and Mat. Trans., **27B**, p.203, (1996).
3. H. Zhang and R. M. German: Int. J. Powder Metall., **38[1]**, p.51, (2002).
4. A. L. Sozinov and V. G. Gavrilijuk: Scripta Mater., **41[6]**, p.679, (1999).
5. M. W. Wu, K. S. Hwang, H. S. Huang, and K. S. Narasimhan, Metall. Mater. Trans. A, in press.