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Effect of Wax Addition on Monel Synthesis by High Energy Milling

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ABSTRACT The effects of addition of polyethylene wax on the synthesis of Cu-Ni alloy (Monel) with 50wt%Ni processed in a high energy ball mill were investigated by X-ray diffraction. The Monel powder was characterized by laser particle size measurement and scanning electron microscopy. The results showed that the addition of wax at the beginning of milling causes a delay in alloy formation, and the particle shape obtained was essentially flaky. However, a strong effect was observed on the shape and particle size distribution when wax was added to the Monel powder with a flaky shape previously processed without wax. The particle shape changed from flake to more irregular and the median particle size decreased from 55 μm to 7.5 μm after 4 hours of milling.

INTRODUCTION

The mechanical alloying (MA) technique is used to synthesize metal alloys in high energy mills from the elementary powders [1]. In this process, the grinding components transfer a great amount of energy to the powder by welding and fracture cycles, giving to the processed material a high chemical homogeneity [2].

The Cu-Ni system was chosen in this work because it presents an isomorphous equilibrium diagram, in which the components are completely soluble in both solid and liquid states. This system presents a wide range of solidification temperatures and accentuated segregation [3]. Nevertheless, ductile powders, as Cu-Ni alloys, when processed in attritor type mills, they tend to present a flaky shape. In general, these flake particles raise difficulties to later conformation, due to a reduction of the compactability and the green toughness of the pieces. To minimize this problem, some authors [4, 5] take an advantage of process controlling agents, in order to modify the particles shape and size of aluminum alloy powders.

In this paper is studied the formation of the Cu-Ni alloy by the mechanical alloying technique and the influence of wax addition in the alloy formation and particle size and shape.

EXPERIMENTAL PROCEDURE

Commercial grade copper and nickel powders with 60 μm and 25 μm average particle sizes, respectively, 50 wt% / 50 wt% mixed, have been used as starting materials. Batches of 100 g mixtures have been processed inside 0.25 l high density polyethylene container in a Netszche (Molinex model) attritor. The following parameters have been defined: ball to powder ratio of 9:1, 1400 rpm and 0.3 ml/min nitrogen flow. Processing time were 1, 2, 3, 5, 7.5, 10, 12.5 and 15 hours.

Another experimental sequence has been followed to study the effect of 1 wt% wax addition to the starting powders. The processing sequence was the same as the one followed for material without wax.

The change in shape and average particle size of monel powders have been evaluated in two ways: first for powders after 5 h attritor processing (high strain hardened state), and second after processing the powders for 5 h followed by 900°C/1h annealing under hydrogen and further disaggregation in attritor for 1 h. These two powders had 1 wt% addition followed by processing as before, for 0.5, 1, 2, 3 and 4 hours.

The final powders have been observed in a Jeol JXA-6400 Scanning Electron Microscope. Particle size distributions have been determined by sedimentation in CILAS 1064 granulometer laser analyzer.

RESULTS AND DISCUSSION

Alloying. Fig.1a shows X-ray diffraction curves of the first experimental sequence, without wax. The 0 h curve shows the elementary powders simple mixture. Between 1 and 3 hours as can be seen, a continuous diffraction peaks enlargement occurred due to an increase in the mechanical hardening and solid solution forming in the copper/nickel interface, caused by the welding process. The 3 hours curve presents a copper and nickel peaks superposition. After 5 hours processing, notice a total solid solution formation and the presence of the alloy peak.

Fig.1b shows the X-ray diffraction curves of the experimental sequence processed with wax addition. The comparison between the two series of curves indicate that the wax addition caused a delay in the alloy formation , being the formation time only after 7.5 hours processing.

One of the alloying mechanisms in the high energy mills is based on the diffusion process helped by the deformation. At first, the great amount of crystalline defects associated to the increase of the temperature due to the particles collisions, helps the interface diffusion. In this way, the number of welding events is a conditioner factor of the alloying kinetics. In the high energy milling process, welding or fracturing events can be predominant. Nevertheless, an equilibrium is reached after some time of processing. Considering this type of mechanism, the wax addition caused a disequilibrium between the particles welding and fracturing, hampering the welding.

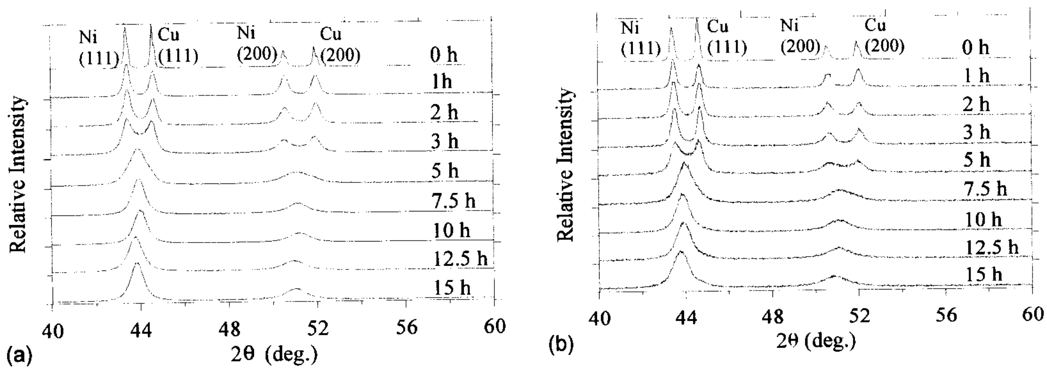
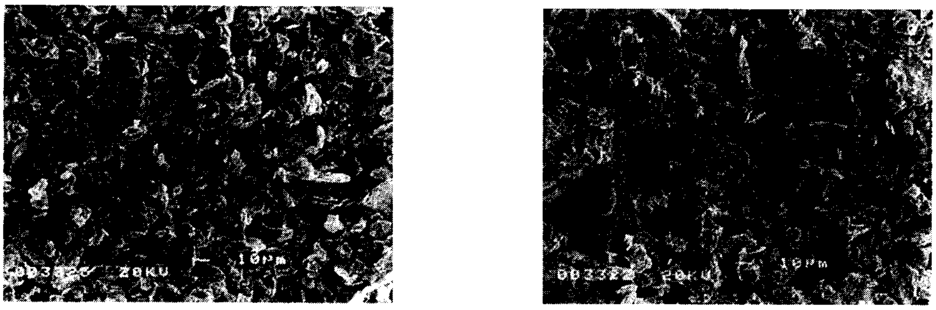


Figure 1: X-ray diffraction patterns of powders processed in high energy attritor mill for times between 1 and 15 hours. (a) without wax. (b) with wax. The 0h curves are related to elemental powder mixtures.



(a) (b)

Figure 2: SEM image of the powder processed for 5 hours without wax(a) and for 7.5 hours with wax(b). The shape of powder particle is essentially like a flake in both cases.

The difficulty in the particles welding process is related to two main causes. Fist, the wax presence acts as a physical barrier between particles. The second is associated with the amount of energy necessary to weld, where the wax being a low melting point polymer absorbs part of the energy.

Shape and Particle Size. Fig.2 shows the particles shape after alloying, 5 hours processing without wax and 7.5 hours with wax. In both cases, can be seen particles predominantly lamellas with average size of 18 and *25* respectively. The wax addition did not affect significantly the particles shape after the alloy formation. Probably this behavior is related to a decrease of the amount of wax in the particles surfaces due to a wax decomposition followed by the absorption of it by the particles. Considering that the statement above is true, the disequilibrium between welding and fracturing presents at the beginning of the process would be progressively diminishing up to redressing the balance. From there on the behavior of both, with and without wax, would be similar, that explain the particles size and shape similarity.

Fig.3 shows the x ray diffraction curves of the starting powders in the conditions I and II. As can be seen in the condition II, the peaks width decrease after the annealing and disaggregation that characterize two different conditions in terms of the mechanical hardening degree.

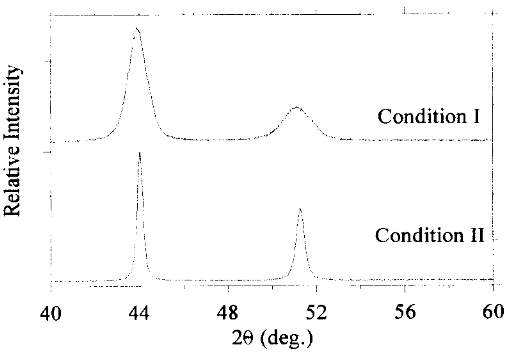


Figure 3 - X-ray diffraction patterns of Cu-Ni alloy powders used as starting powders for the second sequence of experiments. (I) strain hardened powder. (II) annealed and disaggregated powder.

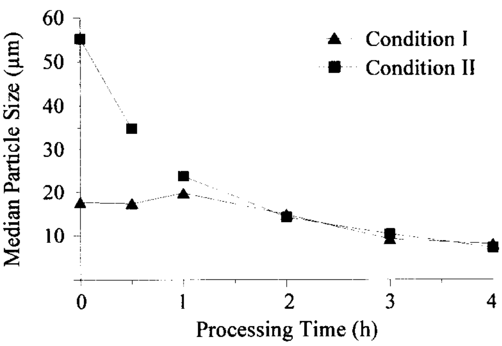


Figure 4 - Median Powder size versus processing time for condition I and condition II.

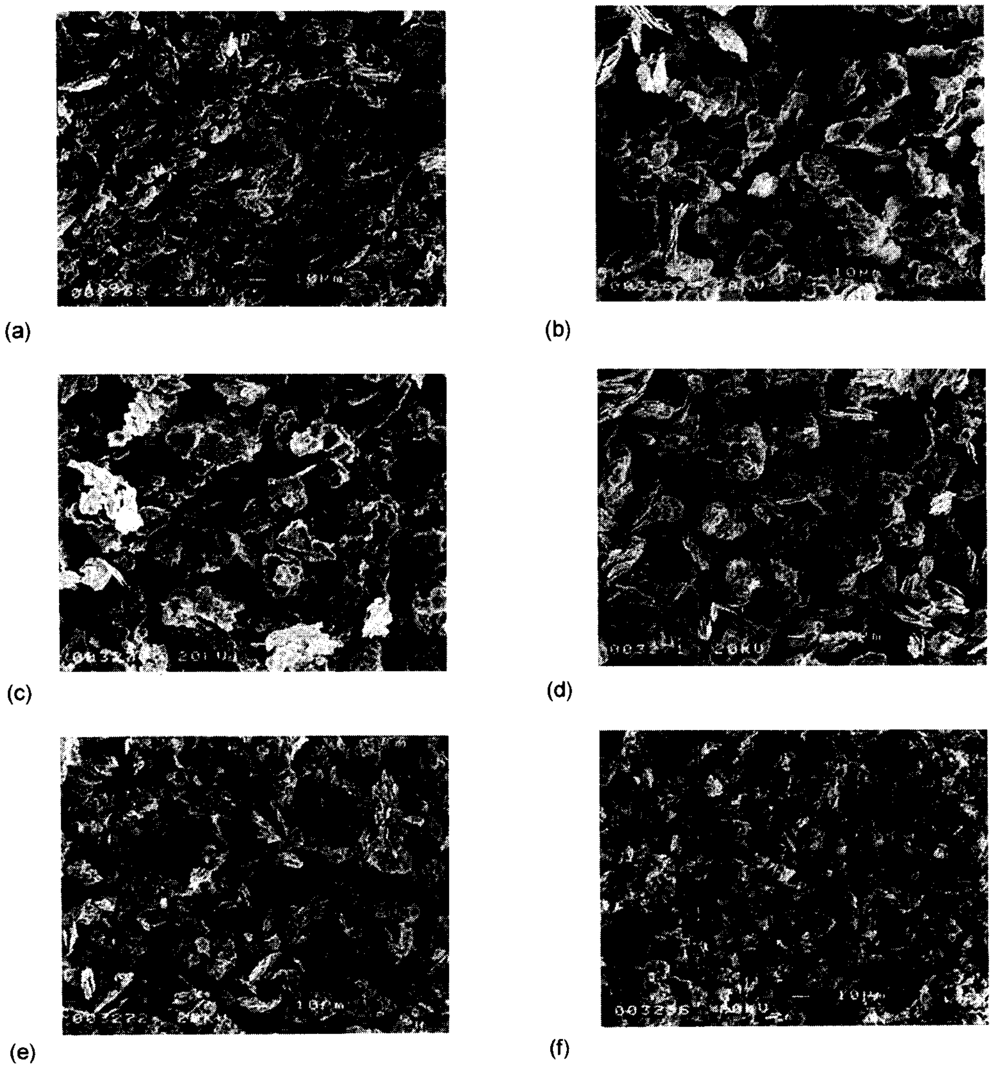


Figure 4 - Scanning electron micrographs show the shape of Monel particles after various processing time for condition I. (a) 0h; (b) ½h; (c) 1h; (d) 2h; (e) 3h; (f) 4h.

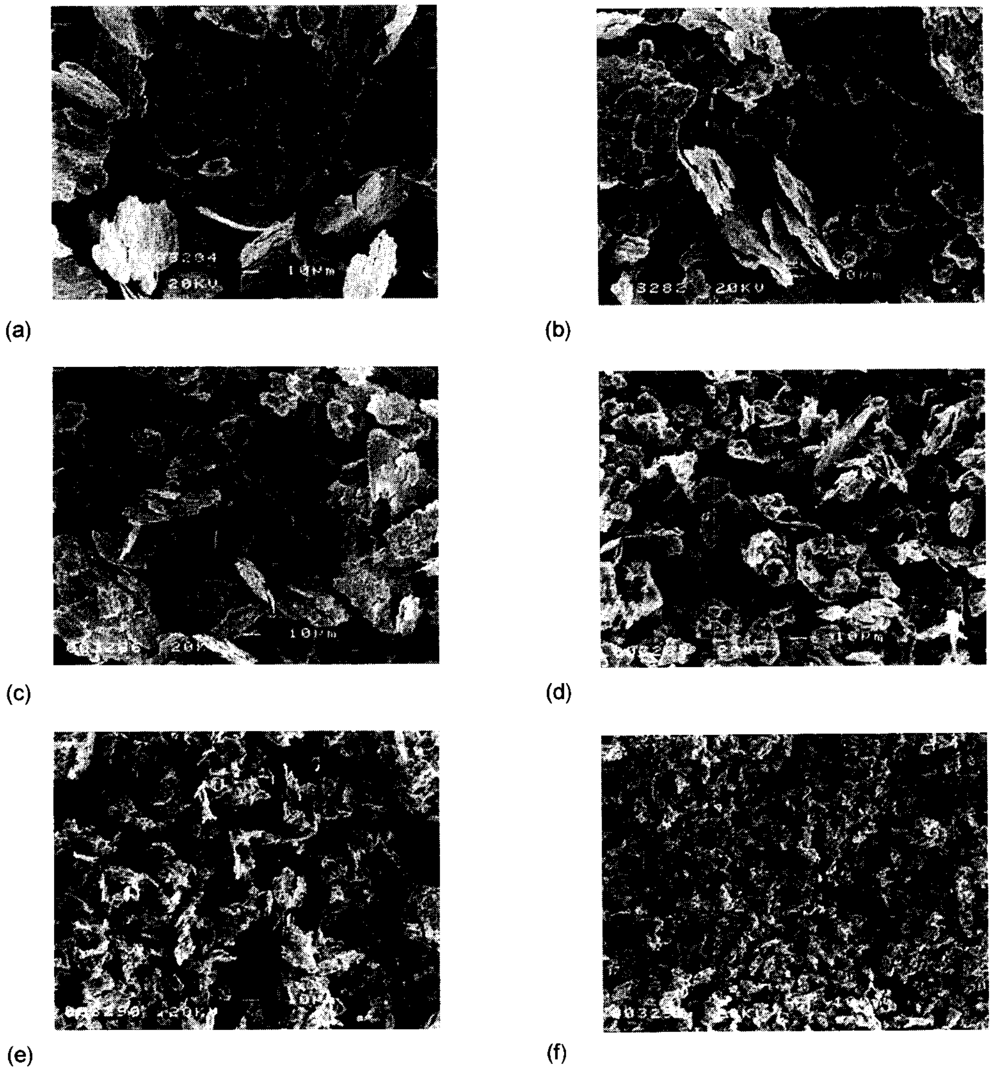


Figure 5 - Scanning electron micrographs show the shape of Monel particles after various processing time for condition II. (a) 0h; (b) 0.5h; (c) 1h; (d) 2h; (e) 3h; (f) 4h.

Fig.4 shows the average particle size as a function of processing time. In condition I, high mechanical hardening, a small increase in the particle size was observed up to 1 hour due to particle agglomeration caused by wax heterogeneous distribution at the initial stage of processing. After 2 hours, a diminishing tendency was observed. In condition II, a diminishing tendency in particle size was observed since the beginning of processing. Despite the starting powders being in different conditions (I and II), after 2 hours of processing, a similar behavior was observed, reaching in both conditions a final particle average size of 7.5

Figs.5 and 6 show scanning electron photomicrographs of the processed powders, where photos 5a and 6a present the particles shape in conditions I and II. The annealing and disaggregation processes caused a significant change in particles size. This behavior is related to the recrystallization and recovery mechanism due to the annealing treatment. The recrystallized particles are less susceptible to fracture during the disaggregation stage, causing again a non-equilibrium between fracturing and welding with a consequent growth in the particles size.

The others micrographs showed in Figs.5 and 6 present the results in agreement with the particle average size measurements. For both conditions, 3 and 4 hours processing showed a tendency for change in particles shape, from lamella to irregular.

CONCLUSIONS

-The alloy was produced after 5 h processing by attrition milling the starting materials. The majority of the particles was found to have flaky shape.

-The addition of polyethylene wax during processing caused a non-equilibrium between particles fracture and welding, turning the welding difficult. As a consequence, there was a delay in the alloy formation.

-By adding wax to the already processed powder having flake particles, the enhancement of the fracture events yielded a large degrease of average particle size, besides a trend to change the particle shape from flake to more irregular.

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REFERENCES

[1] A. Calka, A., Key Eng. Mat., 17 (1993) p. 81-83.
[2] F. Cardelline, G. Mazzone, M. Vittori Antisari, Acta Metall. Mater., 44, 4 (1996) p. 1511-1517.
[3] R. A. Nogueira, A. F. Padilha, Z. Metallkd., 88, 3 (1997) p.204-208.
[4] A. Calka, A. P. Radlinski, B. W. Ninham, , W. A. Kaczmarek, Mat. Sci. Eng., 134A (1991) p. 1346-1349.
[5] J. A. Rodríguez, J. M. Gallardo, E. J. Herrera, J. Mat. Sci., 32 (1997) p. 3535-3539.

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