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Electrical conductivity and microstructure by Rietveld refinement of doped Cu-Ni powder alloys

A. S. Fambrini, W. A. Monteiro, R. M. M. Orrego, I. M. Marques[b],
Juan A. G. Carrió[a]

[1]Physics Department, CCH, Presbyterian Mackenzie University, Rua da Consolação, 896, prédio 12, Consolação, Zip Code 01302-907, São Paulo - SP, Brazil

[a]jgcarrio@mackenzie.br, [b]iara_m_@hotmail.com

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This work presents a comparative study of microstructural and electrical properties of polycrystalline material from two different Cu-Ni alloys: Cu-Ni-Pt and Cu-Ni-Al. The first one of them was produced in electric furnace with voltaic arc and the other was produced by powder metallurgy. The microstructure of the samples was studied by optical microscopy, Vickers micro hardness and x rays powder diffraction. Their electrical conductivity was measured with a milliohmeter Agilent (HP) 4338B. Refinements of the crystalline structure of the samples were performed by the Rietveld method, using the refinement program GSAS. The refinement results and Fourier differences calculations indicate that the copper matrix structure presents not significant distortions by the used amounts of the other metal atoms. In both cases a sequence of thermo mechanical treatments was developed with the intention of increasing the hardness maintaining the electrical conductivity of the alloys. The refinements also allowed a study of the dependence of the micro-structure and the thermo mechanical treatments of the samples. Acknowledgments: Mackpesquisa, CAPES.

Introduction

Microstructural characteristics such as the size, type, form and regular distribution of precipitations determine the mechanical resistance in metallic alloys. This distribution is also fundamental to maintain a high electrical conductivity I the case of the copper alloys. It is also well known that in disordered solid solutions, the resistivity of metals and alloys is strongly influenced by the atomic displacements, vacancies and intersticials. In this work, were produced samples of alloys of Cu-Ni-Pt and Cu-Ni-Al from high purity precursors, to perform a comparative study of the microstructures and physical properties obtained by conventional and powder metallurgy. The alloy elements are added to copper with purpose to improve its resistance, ductility and thermal stability, without causing considerable costs on its form, electric and thermal conductivity and its resistance to the corrosion, typical characteristic aspects of pure copper [14-18]. Products based on copper alloys such as porous material filters, electric friction equipments, contacts and structural parts can be manufactured through the process of powder metallurgy, which have the advantages of making fine grained homogenous structures, forming complicated shapes with close dimensional tolerances and the ability to produce parts with a superior surface finishing [1-13]. These advantages reduce or eliminate costly machining processes and allow less scrap loss, compared to other forming methods. To increase the strength, ductility and formability keeping good electric conductivity of these alloys, there have been used special thermal treatments, as well as variations in the chemical composition.

Experimental Procedure

A Sample of the alloy $Cu_{99,33\%}$ $Ni_{0,23\%}$ $Pt_{0,43\%}$ was produced from high purity precursors, using an electric furnace with voltaic arc in vacuum. The sample was treated at 1073 K during 10 hours. For the preparation of $Cu_{98\%}Ni_{1\%}Al_{1\%}$, high purity powders of copper, nickel and aluminum were mixed for a suitable time and then compacted under 1000 kPa in a cold uniaxial pressing. Afterwards, the specimen was sintered at 800^{o}C in a high vacuum Carbolite furnace that had a hot zone of about 150 mm under vacuum. At last, the sample was homogenized at 550^{o}C under vacuum for 8 hours. For the metallographic and hardness characterization both samples were cold mounted and the compacts were grinding SiC papers followed by fine wet wheel polishing with diamond paste. Vickers hardness of the polished specimens was measured on a HXD 1000TM (Pantec) hardness tester with a load of 100g. Acidic $FeCl_3$ was used as the etchant. The microstructures of the etched samples were observed in an optical microscope. X rays powder diffraction data were collected with a conventional Rigaku MultiFlex diffractometer with a fixed monocromator. The experimental conditions were: 40 kV, 20 mA, $10^o \leq 2\theta \leq 120^o$, $\Delta 2\theta = 0.02^o$, $\lambda_{CuK\alpha}$, divergence slit = 0.5^o, reception slit = 0.3 mm and step time 5 s. The electrical conductivity was measured using an Agilent 4338B milliohmmeter.

Results and discussion

As showed in the optical micrographs of $Cu_{99,33\%}$ $Ni_{0,23\%}$ $Pt_{0,43\%}$ (Figures 1) precipitates are fine and bulk distributed. This influences an increasing of hardness (in relation to pure copper), which resulted in 815,00±0,01 MPa. Pt is 100% soluble and increases 0,635 μΩcm for each 1% of mass increasing. For this sample the measured conductivity was 57 % IACS. The $Cu_{98\%}Ni_{1\%}Al_{1\%}$ sample presents fine grained presences but inadequate porosity due to still insufficient homogenization treatments. This is the reason for the inferior values of hardness and conductivity: 370,00±0,01 MPa and 30% IACS respectively. Further detailed results for powder metallurgy samples are to be published elsewhere. The Rietveld refinement results for $Cu_{99,33\%}$ $Ni_{0,23\%}$ $Pt_{0,43\%}$ indicate that the amount of Ni and Pt for this alloy produced no detectable anisotropic strain or crystallite size effects, as well as no detectable preferred orientation. Otherwise a very slightly broadening of peaks as well as preferential orientation was detected in the $Cu_{98\%}Ni_{1\%}Al_{1\%}$, with GW an order of magnitude greater and a texture index of 1,0062. The asymmetry of the profile function at low angles in both cases is instrumental conditioned. In Tables 1 and 2 are presented the refinement results and profile parameters obtained using function 4 of GSAS.

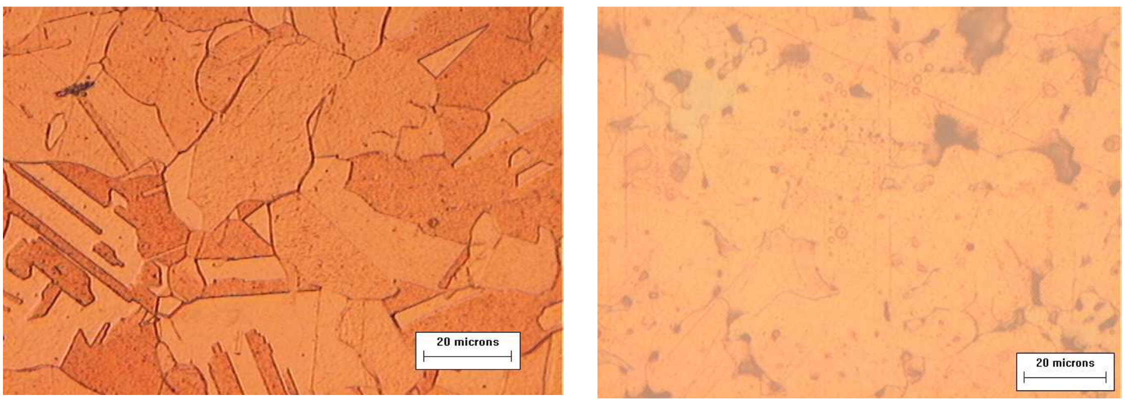

Figure 1: Microstructure of $Cu_{99,33\%}$ $Ni_{0,23\%}$ $Pt_{0,43\%}$ (left) and $Cu_{98\%}Ni_{1\%}Al_{1\%}$ (right).

Table 1: Rietveld refinement results for $Cu_{99,33\%}$ $Ni_{0,23\%}$ $Pt_{0,43\%}$ and $Cu_{98\%}Ni_{1\%}Al_{1\%}$.

$Cu_{99,33\%}$ $Ni_{0,23\%}$ $Pt_{0,43\%}$	$Cu_{98\%}Ni_{1\%}Al_{1\%}$
Cu atomic positions x, y, z: 0.0 0.0 0.0	
Thermal U_{iso} = 0.00957(15) (Å^2)	Thermal U_{iso} = 0.00348(32) (Å^2)
Cell Parameter: a = 3.6169(2) Å	Cell Parameter: a = 3.6174 (7) Å
R_{wp} = 6.92% R_p = 5.20%	R_{wp} = 11.01% R_p = 8.23%
χ^2 = 1.708% R_{Bragg} = 3.27%	χ^2 = 2.532% R_{Bragg} = 5.37%

Table 2: Refined profile parameters for $Cu_{99,33\%}$ $Ni_{0,23\%}$ $Pt_{0,43\%}$ and $Cu_{98\%}Ni_{1\%}Al_{1\%}$ using function 4 of GSAS.

Par.	$Cu_{99,33\%}Ni_{0,23\%}Pt_{0,43\%}$	$Cu_{98\%}Ni_{1\%}Al_{1\%}$
GU	6.29411E+02	1.181E+03
GV	-7.20612E+02	-7.967E+02
GW	2.20778E+02	1.879E+02
GP	0	1.769E+02
LX	0.860743	0
ptec	0.417544	0
trns	-7.21046	9.658E-02
shft	1.64172E+01	3.336E-01

Par.	$Cu_{99,33\%}Ni_{0,23\%}Pt_{0,43\%}$	$Cu_{98\%}Ni_{1\%}Al_{1\%}$
Sfec	0	0
S/L	3.49220E-02	4.257E-02
H/L	1.89520E-02	1.727E-02
ETA	1	0
S400	0.175904	0
S220	-0.15659	-1.591

The adjustments of both diffractograms are showed in Figures 2 e 3 and the more detailed Figure 4 shows the instrumental conditioned asymmetry of the profile function at low angles, which influenced the profile parameters, due to the higher intensity of reflection (111).

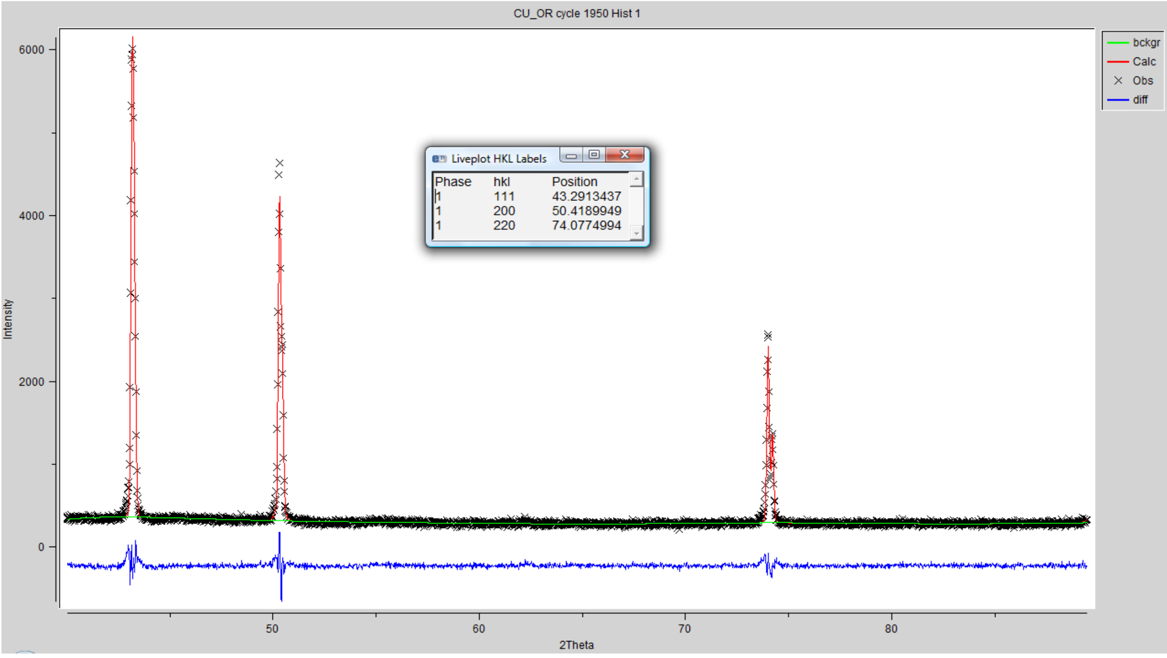

Figure 2: Adjustment of diffractogram for $Cu_{99,33\%}$ $Ni_{0,23\%}$ $Pt_{0,43\%}$

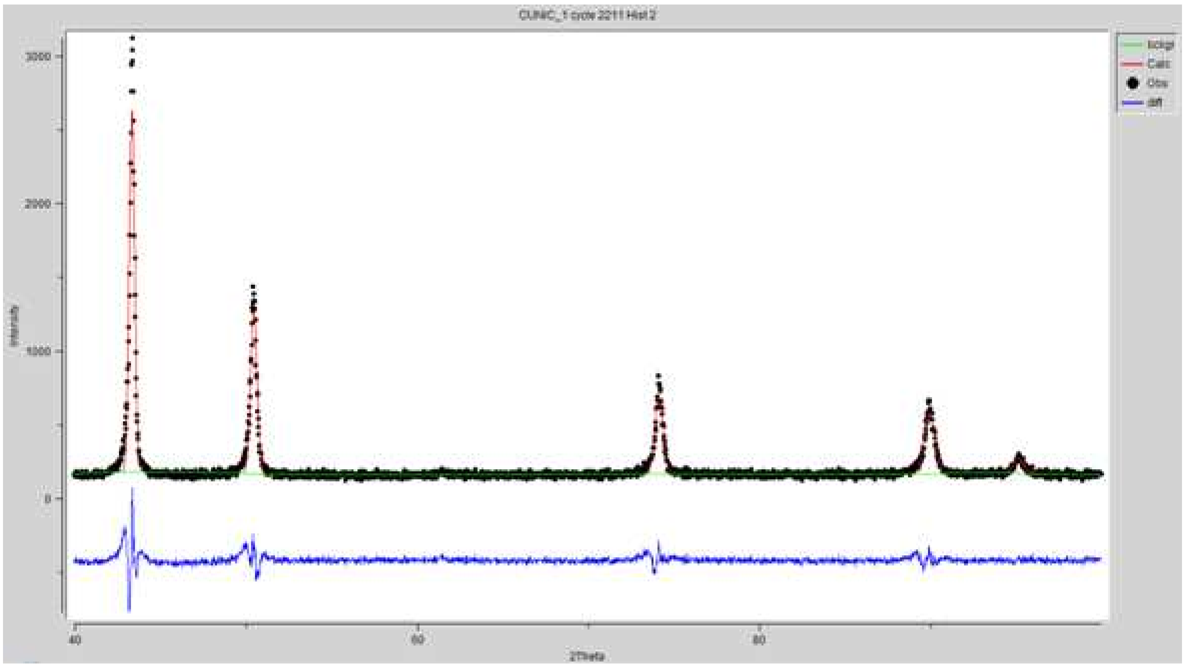

Figure 3: Adjustment of diffractogram for $Cu_{98\%}Ni_{1\%}Al_{1\%}$

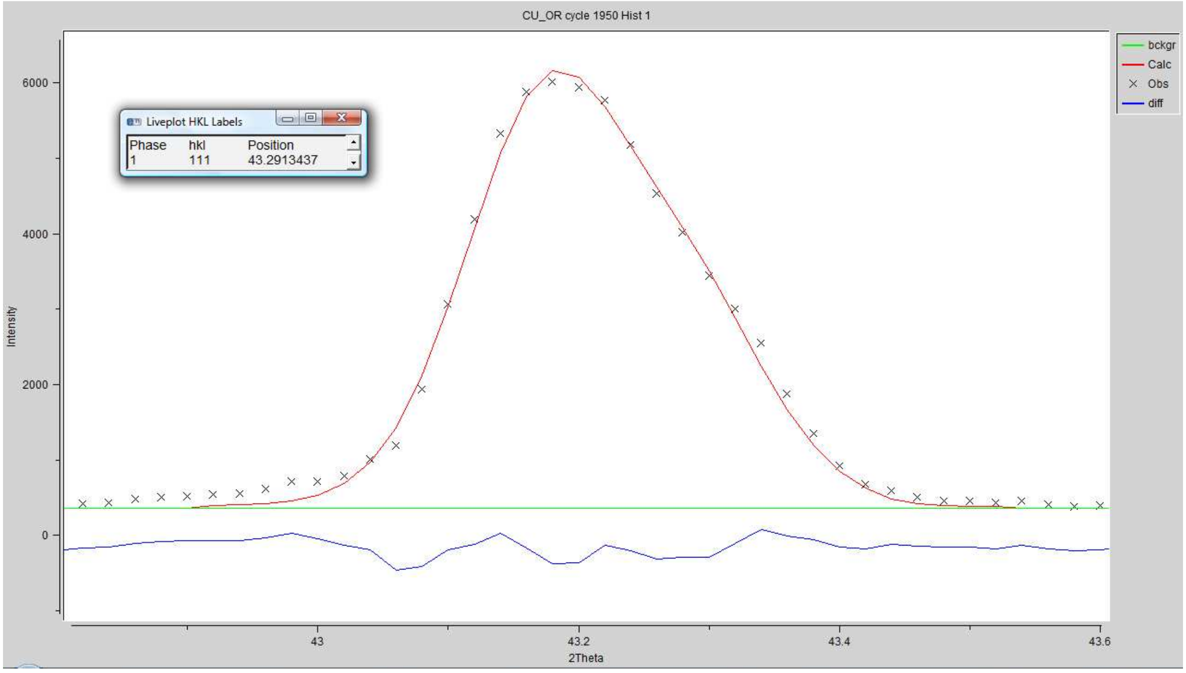

Figure 4: Asymmetric low angle peak profile of Cu (111) reflection of $Cu_{99,33\%}Ni_{0,23\%}$ $Pt_{0,43\%}$.

Conclusions

The obtained microstructure and first-rate mechanical strength (370 MPa) and electrical conductivity (30 %IACS) values for $Cu_{98\%}Ni_{1\%}Al_{1\%}$ could justify a good quality employment for these alloy using powder metallurgy instead conventional metallurgy processing. However, fine grained homogenous structures should be still achieved with further homogenization. Preferential orientation could be a factor to take into account in some cases, as a result of mechanical processing of the powder samples. The small amounts of dopants in both samples caused no structural distortions that could be detectable by x rays powder diffraction. The refinements indicate a very slightly effect in the microstructure of the powder metallurgy sample, possibly due to thermo mechanical processing.

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