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Low Temperature Effect in Electrical Properties of Sintered Copper-Nickel-Aluminum Alloys

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Abstract. The major effort of sintered metallic alloys (compression, homogenization and sintering of metallic powder) is the observation of the evolution of electrical conductivity, mechanical properties (microhardness tests) and microstructures changes after appropriate thermomechanical treatments with the use of copper -nickel-aluminum alloys as electric material. In this case, the purpose was to verify the possible changes in these materials when subjected at low temperatures. Samples of Cu_{x%}Ni_{y%}Al_{z%} initially compressed, sintered and homogenized were characterized by optical metallography (microstructure) and mechanical strength (hardness Vickers) at room temperature. Data of x-ray diffraction of polycrystalline samples were collected with a conventional Difractometer. After this was made measurements of electrical properties (electrical conductivity) at low temperatures of samples obtained from precursors of high purity in powder form, for the study of the influence of powder metallurgy processes in physical properties of metallic alloys in this condition.

Introduction

The aim of this work is to obtain metallic alloys with high mechanical strength and high electric conductivity after adequate optimization of sintering and thermal treatments followed by structural, microstructural, electrical and mechanical characterization of Cu-y%Ni-x%Al alloys (where x and y are variable values). Diverse types of products based on copper alloys can be manufactured through the process of powder metallurgy: porous material filters, electric friction equipments, contacts and structural parts. The alloy elements are added to copper with purpose to improve the resistance, the ductility and the thermal stability, without causing considerable costs on its form, electric and thermal conductivity and resistance to the corrosion, typical characteristic aspects of pure copper [1-12].

The mechanical resistance in metallic alloys depends on the precipitation distribution to obtain similar electrical conductivity comparing to the copper matrix. To increase the strength, ductility and formability keeping good electric conductivity of these alloys, have been used special thermal treatments as well as variations in the chemical composition. In general, the main production of metallic materials is purchased by casting. The contribution to the production of metallic parts by powder metallurgy is increased of consistent outline, supported for enormous return [13-17].

The first one of the advantages is the cost reduction. This project looked for to acquire steadily, in laboratory scale, copper-nickel-aluminum alloys production by powder metallurgy, concerning the maintenance of the electric and mechanical properties with the purpose of getting electric connectors of high performance or high mechanical damping.

The process of making powders, compact them into useful shapes and then sintering them is costly, but the finished parts have some specific advantages over wrought or cast parts. The main advantages are: the possibility to make fine grained homogenous structures; the ability to form complicated shapes with close dimensional tolerances; the ability to produce parts with a superior surface finish. Costly machining processes are thus reduced or eliminated and consequently there is less scrap loss compared to other forming methods [14-17].

All rights reserved. No part of contents of this paper may be reproduced or transmitted in any form or by any means without the written permission of Trans Tech Publications, www.ttp.net. (ID: 200.19.92.200-08/06/15,22:15:24) It is therefore most economical to use powder metallurgy for the high volume production of small, intricately shaped, and/or very precise parts such as gears and links. In addition, the process offers the potential to produce a wide variety of alloys with different material properties such as high temperature toughness and hardness. As the density of the compacted and sintered part influences its key properties of strength, ductility and hardness, a specific porosity is critical. For process control, metallography is used to check porosity, non-metallic inclusions and cross-contamination.

Powder production and mixing is a highly specialized and complex process which produces custom made powder mixes designed to satisfy the needs of a specific application. A good powder mix not only has the ability to produce the required properties of a specific alloy, but also needs to facilitate handling, compacting and sintering. For instance, the easy flow of powder and its capability to mix evenly with other powders is important for an even powder distribution before pressing, and ensures uniform properties of the finished part.

It presents good electrical conductivity of 0.593 $(\mu\Omega cm)^{-1}$ a 300K whereas in the temperature of 77K the conductivity of pure copper goes to 5.263 $(\mu\Omega cm)^{-1}$ demonstrating its excellent application in electric conductors in an ample interval of temperature [18, 20].

The object in this work has been to produce for powder metallurgy ternary copper alloys, CuNiAl, with good mechanical properties and electrical conductivity for use in electric connectors of high mechanical performance in one determined range of temperature. The alloys elements are added to copper aiming at improvement of the resistance mechanics, ductility and thermal stability, electrical and thermal conductivity, beyond resistance to the corrosion.

In temperatures of the order of 20K the copper alloys even continue keeping excellent mechanical resistance [19]. In such a way, it enters the explored aspects in the works get together the electric properties of products gotten for use in low temperatures. The resistivity of copper, as of all the pure metals, varies with the temperature. The conductivity at 373K is equivalent at 80% of the conductivity at 293K. The electric properties also are modified in low temperatures [20].

Thus, beyond the studies of the mechanical and structural properties, they had been initiated measured of electric conductivity to the temperature of 293K, 273K, 195K and 77K aiming at a prospection of these properties in CuNiAl alloys obtained by powder metallurgy. These general characteristics had been detected in mechanical tests in fifteen different studied copper alloys, including the commercially pure brass, bronzes and coppers. They had been tested for the brass industry to verify properties such as tensile strength, Young's modulus and impact in temperatures up to 4 K for pure copper. The resistivity in metals and alloys is strongly influenced in disordered solid solutions for the atomic displacements, interstitial defects and vacancies [18, 19].

The characterization techniques utilized in this work: structural characterization (R-X diffraction), microstructural analysis, measurements of electrical conductivity and mechanical properties of the CuNiAl alloys.

Experimental Procedures

This project looked adequately for to acquire knowledge, in laboratory scale, obtaining copper-nickel-aluminum alloys with powder metallurgy processing, aiming the electric and mechanical properties maintenance, to get electric connectors of high mechanical performance or high damping that can be used in a reasonable temperature range.

For the production of components the mixed powders are first compacted under high pressure in a suitable system. At this stage the part has the geometrical features of the finished component, but not its strength and is called the "green" part. The bonding occurs through diffusion between adjacent particles. In order to develop the mechanical and physical properties of the material, metallurgical bonding has to take place through sintering at high temperature in a sintering furnace. The bonding occurs through diffusion between adjacent particles. To avoid oxidation, which would impair the interparticle bonding, the sintering process is conducted in a protective atmosphere or convenient high vacuum. The bonding increases the density, and pressed and sintered powder metal parts generally contain some residual porosity.

The as-pressed compacts were conventionally sintered in a high vacuum Carbolite furnace that had a hot zone of about 150mm. The Cu-Ni-Al alloys can be consolidated by solid state sintering. The most important conditions are presented in Table 1.

In research and failure analysis, metallography is a major tool used to develop new products and improve manufacturing processes. In addition to chemical analysis, quality control also includes physical methods for checking density, dimensional changes, flow rate etc.

| Condition | metallic powder premixed | | | | | |
|---|---|-----------------------|----------------------|--|--|--|
| Compaction pressure | 50 MPa | | | | | |
| Chemical alloy | Cu-0.5%Ni-0.5%Al; Cu-1%Ni-0.5%Al; Cu-1%Ni-1%Al; | | | | | |
| composition (wt %) | Cu-3%Ni-3%Al; Cu-5%Ni-5%Al | | | | | |
| Sample dimensions | Cylinder diameter $\phi = 10.2 \times 10^{-3} \text{ m}; \text{ h} = 14.8 \times 10^{-3} \text{ m}$ | | | | | |
| Sample weight | 6.5x10 ⁻³ kg | | | | | |
| Sintering temperature and conditions | Sintering | premixed condition | Vacuum | | | |
| | temperature (K) | premixed condition | pressure | | | |
| | 023 1073 | Solid state sintering | $1,3 \times 10^{-3}$ | | | |
| | 923 - 1073 | Solid state sintering | milibar | | | |
| Sintering time | $t_{\rm S} = 1.8 \times 10^3$ a 5.4×10^3 s | | | | | |
| Homogenization time (s) | $t_{\rm H} = 3.6 {\rm x} 10^2 {\rm a} {\rm 28.8 {\rm x} 10^2}$ | | | | | |

Table 1 - Sintering parameters of Cu-Ni-Al

Cold mounting of the sintered and homogenization sample was done by optical and hardness studies .The compacts were grinding with 400, 600, 800, 1000 and 1200 SiC papers followed by fine wet wheel polishing (diamond or alumina pastes). Vickers hardness of the polished specimens was measured on a hardness tester (load of 100g). Acidic FeCl₃ was used as the etchant. The microstructures of selected etched samples were observed in an optical microscope. Special samples for electrical conductivity studies were utilized an Agilent 4338B milliohmmeter and for crystallographic parameter an R-X diffractometer [21].

Results and Discussion

The Table 2 resume some data realized until now with the samples of copper nickelaluminum alloys concerning mixing, compacting, sintering, homogenizing treatments and also values of hardness and electrical conductivity. The intention is to continue the study of these alloys to obtain the best condition for electrical and mechanical application with powder metallurgy processing.

Table 2 – Some mechanical and electrical properties of the copper-nickel-aluminum alloys obtained by powder metallurgy

| Alloys | Sintering | | Homogenizing | | Mechanical Resistance | |
|----------------|-----------|-------------|--------------|-------------|-----------------------|--|
| (weight %) | T (K) | $t(10^3 s)$ | T(K) | $t(10^3 s)$ | (MPa) | |
| Cu 1.0Ni 0.5Al | | 5,4 | | | (420 ± 20) | |
| Cu 1.0Ni 0.5Al | | | 773 | 21.6 | (280 ± 10) | |
| Cu 1.0Ni 1.0Al | 1053 | | | | (240 ± 20) | |
| Cu 1.0Ni 1.0Al | | | 773 | 32.4 | (370 ± 20) | |
| Cu 5.0Ni 5.0Al | | | 773 | 21.6 | (400 ± 20) | |

Table 3 – Electrical properties at room temperature and at low temperatures of CuNiAl alloys obtained by powder metallurgy

| Alloys (weight%) | Sintering | | Homogenizing | | Electrical Conductivity (% IACS) | | |
|---------------------|-----------|--------------|--------------|--------------|-------------------------------------|----------|-----------|
| | T(K) | $t(10^{3}s)$ | T(K) | $t(10^{3}s)$ | T = 300K | T = 195K | T = 77K |
| Cu 1.0Ni 0.5Al | 1053 | 5,4 | | | (30 ± 5) | (53 ± 6) | (109 ± 8) |
| Cu 1.0Ni 0.5Al | | | 773 | 21.6 | (35 ± 8) | (55 ± 5) | (97±6) |
| Cu 1.0Ni 1.0Al | | | | | (29 ± 7) | (44 ± 5) | (85±7) |
| Cu 1.0Ni 1.0Al | | | 773 | 32.4 | (30 ± 5) | | |
| Cu 5.0Ni 5.0Al | | | 773 | 21.6 | (28 ± 7) | | |

(Observation: conductivity values obtained with electrical resistivity)

The mechanical resistance in metallic alloys depends on the precipitation distribution to obtain similar electrical conductivity of the copper (matrix). To increase the strength, ductility and formability keeping good electric conductivity of these alloys, have been used special thermal treatments as well as variations in the chemical composition. At moment the mechanical strength (400MPa) and electrical conductivity (35%IACS) values indicate a good application for these alloys utilizing powder metallurgy instead conventional metallurgy.



Figure 1 Optical micrograph of the alloy Cu-1%Ni-1%Al, cold compact (50 MPa) and sintered at 650C for 1200s



Fig.2- Optical micrograph of the alloy Cu-1%Ni-0.5%Al, cold compact (50MPa) and sintered at 780°C for 5400s.



Fig.3- Optical micrograph of the alloy Cu-1%Ni-0.5%Al, cold compact (50 MPa) and sintered at 780°C for 5400s



Fig.4- Optical micrograph of the alloy Cu-5.0%Ni-5.0%Al, cold compact and sintered at 780°C for 5400s and also homogenized at 500°C for 21600s.

Concerning the microstructural aspects, figures 1 to 4 show optical micrographs of some Cu-Ni-Al alloys. Fine grained presences but with inadequate porosity and second phases until now show that new homogenization treatments will be necessary to overcome this situation and also investigations with scanning and transmission electron microscopy to identify the presence of second phase on these alloys. The powder diffraction data indicate that the utilized amounts of dopants do not distorted the copper matrix structure significantly. No special broadening of the Bragg peaks was detected, which indicates that crystallite sizes are not affected.

In the studies of electric conductivity was used the milliohmmeter 4338B (Agilent) and to obtaining the crystallographic parameters one rays X diffractometer [21]. The tests of electric conductivity were made at room temperature and also at the temperature of liquid nitrogen to make comparison of the sample in low temperature.

Conclusion

The applied powder metallurgy processing steps on the copper-nickel-aluminum alloys confirm a good mechanical strength (400MPa) and electrical conductivity (35%IACS) values that indicate a good application for these alloys utilizing powder metallurgy instead conventional metallurgy. The possibility to search and make fine grained homogenous structures, the skill to form complicated shapes with close dimensional tolerances and the capacity to produce parts with a superior surface finish with close dimensional tolerances encourage this metallurgical application.

Also in situations that the electrical systems operate in low temperatures, according the results until now obtained. Although not yet conclusive, therefore if it needs specific mechanical testes at low temperatures (impact tests), motivates the continuity of this research. The obtained results showed the conductivity value of 0.650 ($\mu\Omega$ cm)⁻¹. These results must be reaffirmed in the next measures in a new research. The possibility to search and to produce fine and homogeneous structures, the ability to get products with special formats of complicated dimensions and with narrow dimensional tolerance and the capacity to produce parts with a superior finishing, with indices of dimensional tolerance stimulate this technological application.

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