Characterization of microstructural and mechanical properties after cold rolling of an electro-sinter-forged Cu-Sn alloy

Arch. Metall. Mater. **65** (2020), 2, 787-792

DOI: 10.24425/amm.2020.132821

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CHARACTERIZATION OF MICROSTRUCTURAL AND MECHANICAL PROPERTIES AFTER COLD ROLLING OF AN ELECTRO-SINTER-FORGED Cu-Sn ALLOY

Traditional press and sinter processes have gained in the last decades more and more importance in the manufacturing of high volume and precise mechanical components especially in the field of iron based powders. In recent years, the reductions of processing times and temperatures were spotted as critical targets to increase productivity and reduce energy consumption. Electric current assisted sintering (ECAS) technologies have always been seen as an alternative to traditional furnace based sintering techniques and have been the target of different researches with the specific purpose of reducing both operational times and costs. The aim of the present study is to investigate the effect of an innovative process called Electro Sinter Forging (ESF) applied to CuSn15 powders. Thanks to a very short processing time (less than 1 second to densify loose powders), this process is able to retain a very small grain size, thus enhancing mechanical properties of the processed materials. Furthermore, to the authors knowledge, cold – rolled electro – sinter – forged alloys has never been investigated before. First of all, bars were electro – sinter – forged and subsequently characterized in the as sinter – forged condition. The observation of microstructure evidenced an extremely fine microstructure and a reduced degree of porosity. Afterwards, bars were cold rolled after different reduction ratios; macrostructural integrity of the rolled bars was assessed before evaluating the effects of cold rolling on the sinter – forged microstructure.

Keywords: Electro-Sinter-Forging, Powder metallurgy, CuSn15, Rolling

1. Introduction

Powder metallurgy (PM) technologies for the production of parts in both ferrous and non – ferrous alloys have been gaining plenty of attention from the international scientific and industrial community in the last ten years mainly through the propulsive and innovative force of additive manufacturing [1-3]. There are however, in parallel, less known novel PM methods that are rapidly moving their way to industrialization without the same fanfare. The techniques known as electric field assisted sintering techniques (FAST) [4], electric current assisted sintering (ECAS) [5] and particularly the less known range of electric pulse consolidation (ECP) methods [6]. The most recent Electro-Sinter-Forging (ESF) or e-forging method is gaining traction and has demonstrated to be interesting for the markets of diamond metal matrix abrasive tools, precious metal parts, hard metal cutting tools, steels and shape memory alloys [7-11]. Novel applications and uses are emerging thanks to the intrinsic advantages of these methods: simplicity (one machine for forming and sintering to near net shape), lean production, rapidity (cycle time <10 seconds per part), very limited amount of energy employed, and low wear of tooling if compared to conventional casting [12] or cutting techniques [13]. The technical and manufacturing advantages of these techniques combine with the possibility of creating innovative materials such as metal composites and diamond composites with novel, high performance, properties and extremely high densities. The process, named Electro-Sinter-Forging (ESF) is simple: two pulses, one mechanical and one electrical, are superimposed in a die previously loaded with the powders. A schematic representation of the electro-mechanical system used for ESF is given in Figure 1. The electrical pulse is generated by a high voltage capacitor bank and then transformed to a high current – low voltage electro-magnetic discharge and is synchronized in a way that energy is transferred

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after a predetermined level of pressure is reached. In such way a homogeneous flow of current is guaranteed. While the energy source is transferring electro-magnetic energy, the mechanical system acts to compensate the powder shrinkage by increasing the applied pressure. After holding in pressure from a few milliseconds to, usually 1-3 s the powders while consolidating, the upper plunger is automatically drawn out of the die and the lower plunger is moved to the upper part of the die in order to extract the sintered piece from the die assembly. Copper based alloys are widely diffused in power train, automotive, naval sector and depending on the application, the alloys composition can differ significantly. In the frame of this study, a CuSn15 Tin – based bronze (alloy grade UNS 91000) was densified via ESF from gas atomized metal powders (Fig. 2). The batch of CuSn alloy powders employed for this research was admixed with pure Cu powders (Fig. 3) in order to obtain the proper weight ratio between Cu and Sn as for the CuSn15 alloy. Admixing is a common practice in powdered alloy production but to the authors best knowledge there is no investigation in literature on its effects on rapid sintered microstructures such as those obtainable through ESF. For these reasons, an experimental study was performed to characterize CuSn15 admixed alloy from the microstructural point of view, in the ESFed state and soon after cold rolling.

2. Materials and methods

Commercial prealloyed CuSn25 powders (20-60 μm average size) were admixed with pure Cu powders to obtain an overall CuSn15 composition: one kg of CuSn25 powders was poured in the mixer and then 0.4 kg of pure Cu powders were added to reach the final $\%wt_{Sn}$ of approximately 15.0 ± 0.1. The powders were weighted with a Kern PLS 510-3 precision balance and manually loaded through a funnel placed inside a 100 mm long die. The die was previously mounted on a machine for Electro-Sinter-Forging by EPoS (www.eposintering.com) equipped with a press able to reach a force of 120 kN maximum and a generator of electric current able to provide up to 100 kJ of energy to the loose powders. Ten bars, 100 mm long with a 3×4 mm cross – section were then ESFed and 8 of them subjected to cold rolling. After compacting the powders by ESF, the sample density was calculated as a ratio between sample volume to sample weight using Archimedes method. The experimental densities were then compared to the theoretical density for CuSn15 to calculate the final percentage of theoretical density obtained. The theoretical density for CuSn15 was taken as 8.69 g/cm^3 [14].

The rolling reduction ratios employed are reported in Table 1. The electro-sinter-forgings were performed in air,

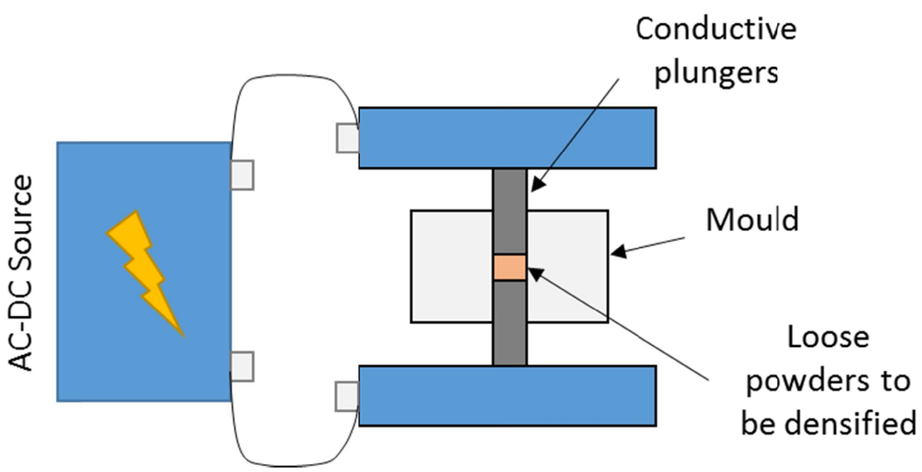


Fig. 1. Schematic representation of an Electro-sinter-forging machine in accordance to the description given in the patent

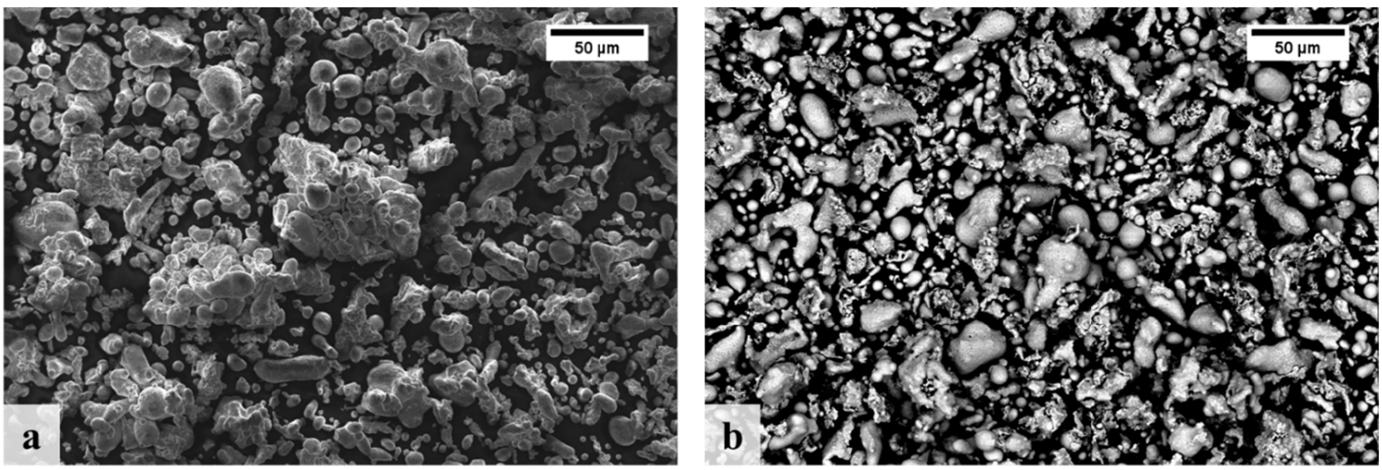


Fig. 2. SEM images of CuSn15/Cu admixed powder batch used for E-forging samples. a) SE image, b) BSE image

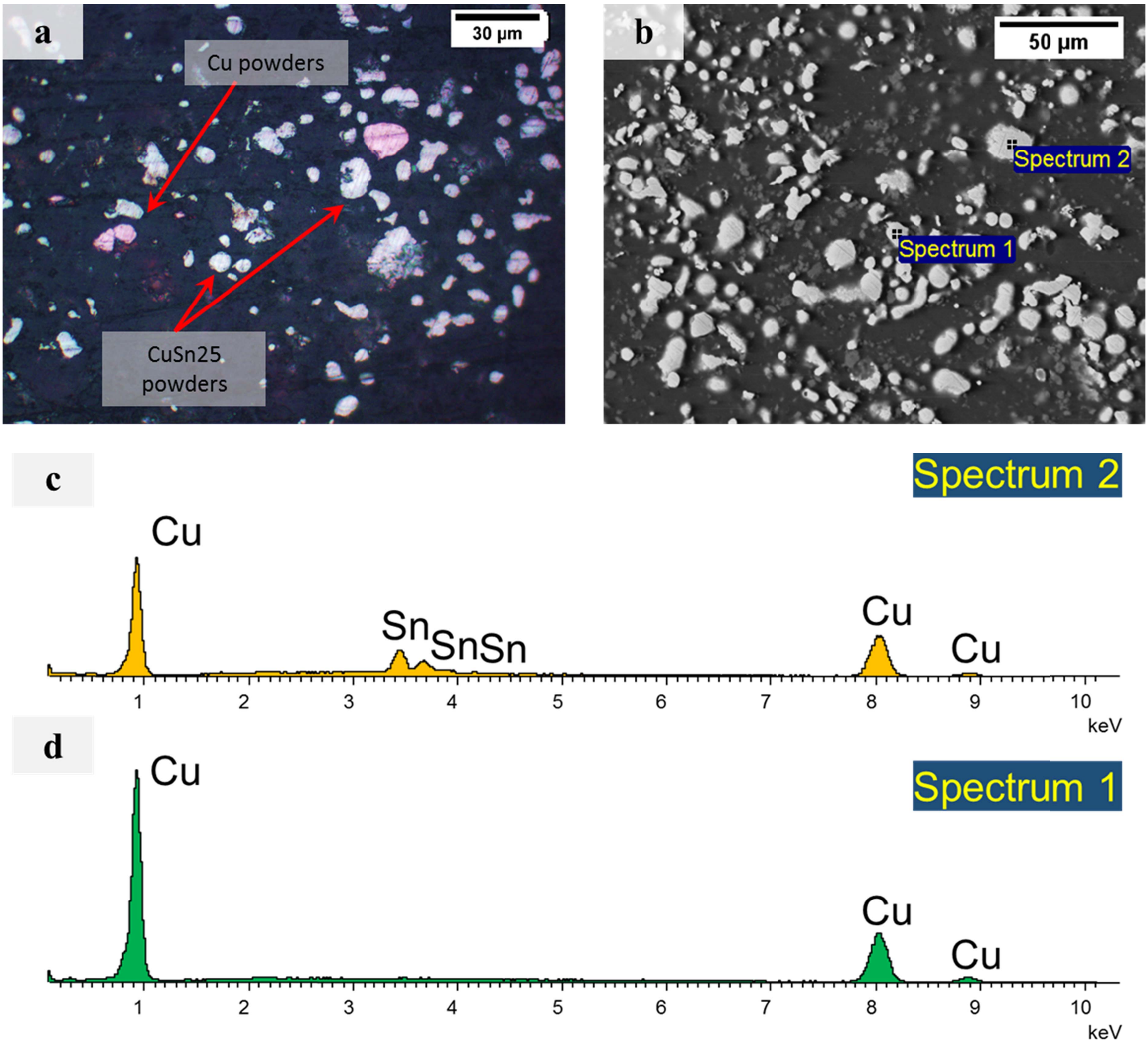


Fig. 3. Overview of powder quality. a) Light optical micrograph, different grades of powders are highlighted. b) Secondary electrons image of polished powders, the points where EDS analysis were performed, are highlighted d) Results from EDS semi – quantitative microanalysis showing a difference in composition between CuSn25 powders and Cu powders

without a control atmosphere, since all past experiences even with highly reactive materials such as Nitinol or titanium, have demonstrated no tendency at all to pick up oxygen during ESF [15]. Oxygen content was measured by Leco ONH analyser for light elements (oxygen, nitrogen, hydrogen) for both powders and electro-sinter-forged samples.

One of the samples was cut and prepared for metallographic observation after electro-sinter-forging while other samples were cold rolled and then observed by light optical microscope (Leica Mef4). The open source software Hugin was used for merging micrographs together to obtain an overview of the whole cross – section of the as – ESFed sample (Fig. 4). Mechanical properties of electro-sinter-forged and electro-sinter-forged + cold rolled samples were characterized by micro Vickers hardness tester

TABLE 1

Design of the rolling experiments, the samples are listed from number 1 to 8 based on the reduction of thickness obtained by cold rolling

Sample [#]	Post rolling thickness [mm]	Thickness reduction [%]
1	3.00	0.00
2	2.95	1.67
3	2.85	5.00
4	2.51	16.33
5	2.38	20.67
6	2.05	31.67
7	1.58	47.33
8	1.16	61.33

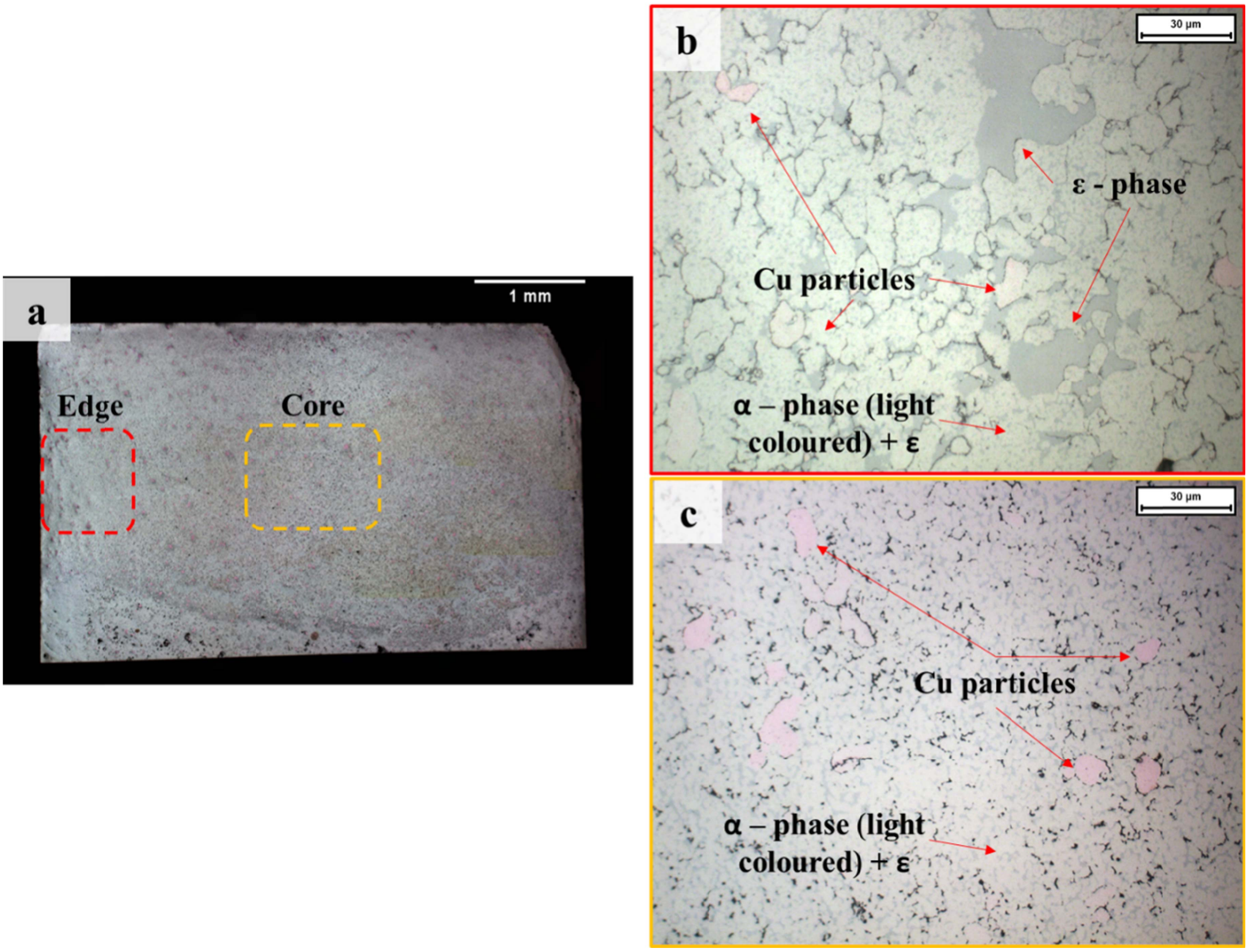
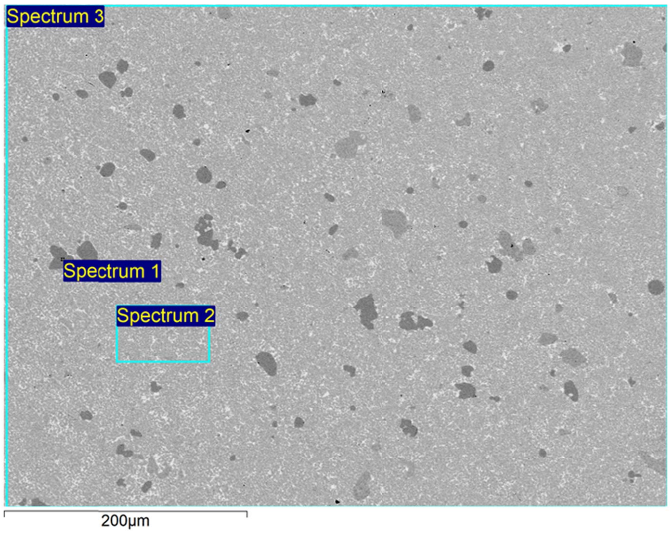


Fig. 4. Micrographs of sample #1 after Electro-Sinter-Forging. a) Merged micrographs to cover the entire sample cross – section, b) microstructure at 500× at the edge of the cross – section, c) microstructure at 500× at the center of the cross – section

(Leica VMHT) with a load of 0.2 kgf. No etchant was used to reveal microstructure because this was evident just after polishing with silica (SiO_2). Both chemical composition and morphology of powders was characterized by SEM and EDS (Leo 1450 VP equipped with Oxford EDS probe for microanalysis).

3. Results and discussion

Electro-sinter-forging of the powders resulted in fully dense sample (>98%) characterized by small isolated pores with an average size lower than 1 µm (0.85 ± 0.13 µm). The negligible Oxygen take up was confirmed by chemical quantitative analysis, no significant increase was detected if compared to the starting powders (0.05 ± 0.008% in the starting powders, 0.06 ± 0.004% in the as sintered parts). The structure after sintering through the cross – sectioned sample is non – uniform, more precisely it is characterized by a core/edge pattern (Fig. 4b,c). The most external part of the sintered sample (edge) retains a coarser microstructure that still has reminiscences of the prior powders shape while the internal part of the sample (core) is much finer. This effect is strongly related to the physics of the electric current assisted sintering and to the current path itself. By observing the microstructure of ESFed sample (Fig. 4), two main phases are detected as predicted by the Cu-Sn phase diagram: αCu and Tin rich ε-phase. Moreover, some bright pink/red grains are evident both in the core and in the edge of the sample. By comparison with Figure 3 it can be supposed that these grains are still pure admixed Cu: this hypothesis is confirmed by EDS microanalysis (Fig. 4). ESF is so rapid and effective to deliver enough energy to densify the starting powders without melting them; powders are compacted and sintered without affecting their chemical composition. The difference in chemical composition between CuSn25 and Cu is still evident if the single microstructural constituents are analysed separately (Sp. 1 and Sp. 2 in Fig. 5) but if a large region of the microstructure is analysed, the average composition is that one of a CuSn15 (Sp. 3 in Fig. 5). No alloying effect of the sintering process was observed during these experiments. Cu powders seem to be totally unaffected, with a particle morphology unaltered if compared to the powdered state (Fig. 3). At this stage ESF appears to be very effective in preserving the fine microstructure proper of powders without the occurrence of large porosities in the as – sintered material. If compared to a conventional technique such as casting, the ESFed microstructure is extremely fine; this can be responsible for the final enhanced mechanical properties through mechanisms such as grain refinement. The hardness of the cold rolled samples increases after cold plastic deformation due to work hardening of the ESFed alloy (Fig. 6). Cold rolled samples were deformed without breaking but cracks on the surface of the samples was more severe with increasing rolling ratios.



	Cu [%wt]	Sn [%wt]
Sp_1	99.71	
Sp_2	74.42	25.58
Sp_3	84.71	15.29

Fig. 5. BS – SEM image of the as-densified microstructure where three specific regions have been analysed

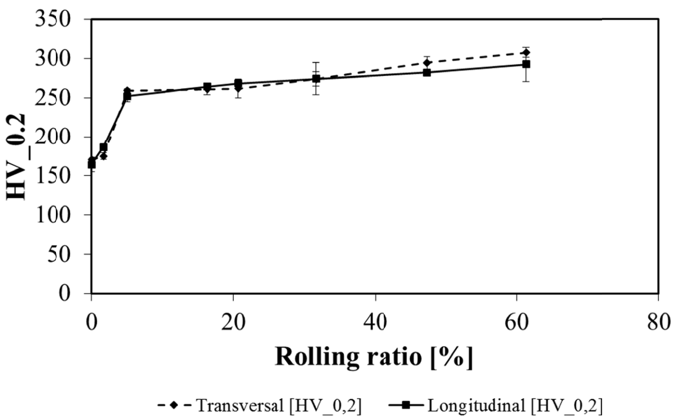


Fig. 6. Influence of rolling ratio on microhardness of ESF CuSn bronze

An innovative electric current assisted sintering method called Electro-Sinter-Forging (ESF) was employed to densify CuSn25 bronze powders with admixed pure Cu powders obtaining dense and consistent bar shaped samples for microstructural and mechanical characterization. The microstructure of the electro-sinter-forged bronze is much finer than conventional casting processes and retains microstructural features proper of powders, which are possibly to compare with other novel techniques such as severe plastic deformation or additive manufacturing [16-20]. The high potential of this technique is based on the rapid heating that can be supplied.

4. Conclusions

In the present study a novel capacitive discharge sintering named electro-sinter-forging has been employed to densify a tin bronze. A CuSn25 alloy was admixed with pure Cu powders and then densified closely to theoretical density (>98%). Pure Cu particles are still present in the densified microstructure after sintering. ESFed CuSn bronze bars were cold deformed without breaking up to 60% and moreover a significate strain hardening effect was detected. It was proved that by a proper selection of the processing parameters it is possible to preserve even pure Cu grains inside of a CuSn alloy where Cu itself is still soluble. From these experimental evidences it can be concluded that complex microstructures are obtainable through ESF. Thanks to its effective combination of ultra – rapid sintering and high final density, innovative metallurgical systems are conceivable. These observations together with currently on-going experimentations on other materials, will serve as guidance for future studies leading to a more complete characterization.

Acknowledgements

Authors are grateful for the support of experimental works by project VEGA 1/0732/16.

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