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# CHARACTERISATION OF THIN Ag AND Cu METALLIC STRIPS

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#### ABSTRACT

In this paper we present production of silver and copper strips. Those strips are use for fusing elements. Metallic strips have to possess the appropriate dimensions with related tolerances, high enough hardness (> 70 HB), specific electrical conductivity (for Ag: >  $61 \pm 2$  Sm/mm<sup>2</sup>, for Cu: >  $58 \pm 2$  Sm/mm<sup>2</sup> at 20°C), optimal physical properties, good mechanical properties with tensile strength higher than 220 N/mm<sup>2</sup>. It is very important to know that all required properties are constancy. The surfaces of the strips have to be free of oxides or any other defects. These demands can be accomplished by controlled thermo-mechanical treatment, including a combination of controlled plastic deformation and heat treatment.

The goals of our research are to study the influence of the processing parameters, during conventional thermo-mechanical (TM) treatment. Parameters of the process effecting on the microstructure and mechanical properties of produced silver and copper strips. The optimisation of the process production technology is in the Slovenian company, Zlatarna Celje. Moreover, to study some alternative technological methods i.e. melt spinning strip production that could enable appropriate, or even new, mechanical properties of the strips in an easier way or at a lower cost.

Key words: Ag and Cu strips, characterisation, physical and mechanical properties

## **1. INTRODUCTION**

Thin Ag and Cu metallic strips are used as elements in safety mechanisms in electro installation commutators. The most important part of all the mentioned devices is the melting element. In the case of electricity this elements protect the other facilities. The melting element is usually a thin metallic strip, treated with special tools thus creating the contractions and cutting-outs of different shapes. The shape of the cut-out is adjusted to the specific demands of the melting element, according to the terms of use; the facility

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of which is to protect: the motor, installations, half conductors etc. Ag metal strips with a high degree of clarity are used in the manufacturing process of melting elements for safety fuses D0, D, NV, VV from 2 to 1250 A, for electrical contact elements and others. In the future there are additional perspective fields of application. Moreover, it can be seen, that in the future there will be the possibility of using Ag strips for the base of super-conductivity elements – strips. The strips of width 1.6 mm are used for high voltage melting safety fuses. Wider strips with width 62 mm are used for Ultra-Quick characteristic melting safety fuses. Cu strips are used as melting elements in safety fuses D0, D and NV from 2 A to 1250 A, and as electrical contact elements etc. The consumption of Cu strips is significantly higher than Ag strips, by approximately factor 10.

The main specific requirements for presented metallic strips are:

- for Ag strips: hardness 45-80 HV; specific electrical conductivity: 58-63 Sm/mm<sup>2</sup> at 20°C; sabre-shape: 1,00 mm/m; required tolerance ± 0,005 mm for the final strip thickness 0,04 mm 0,30 mm; different required tolerances according to the final strip widths: + 0,04 mm for the strips width 1 mm 8 mm, + 0,15 mm for the strips width 8 mm 20 mm, + 0,20 mm for the strips width 20 mm 50 mm, ± 0,30 mm for the strips width > 50 mm;
- for Cu strips: hardness (soft) 35-55HV, hardness (hard) 95-150HV; specific electrical conductivity: 56-58 Sm/mm<sup>2</sup> at 20°C; sabre-shape: 1,00 mm/m; required tolerance ± 0,005 mm for the final strip thickness < 0,30 mm, and different required tolerances according to the final strip widths: + 0,04 mm for the strips width 1 mm 8 mm, + 0,15 mm for the strips width 8 mm 20 mm, + 0,20 mm for the strips width 20 mm 50 mm, ± 0,30 mm for the strips width > 50 mm;

In the production of Cu and Ag strips the most important and common phase is rolling. This process is known as the process of plastically deforming metal by passing it between rolls. In deforming metal between rolls, the work is subjected to high compressive stresses from the squeezing action of the rolls, and to surface shear stresses as a result of friction between the rolls and the metal [1]. The process of strip rolling is convenient for defining the various basic parameters. Furthermore, the sheet is the form of starting material. The characteristics (dimensional accuracy, forming behaviour, and surface quality) and the manufacturing conditions of the sheets are very important for their optimum use in various deformation processes [2, 3]. During the rolling process, the rolls exert compressive stresses on some portions of the work-piece. These stresses cause stresses in the work-piece, resulting in plastic flow. The rolling process also imparts a characteristic texture to the rolled work-piece. The texture affecting the further forming characteristics of the work-piece can be controlled to a certain extent by proper combinations of rolling and heat treatment conditions [4]. In the rolling process there are a variety of problems, leading to specific defects in the finally produced metallic strips. Defects can arise, depending on the interaction of the plastically deformed work-piece with the elastically deforming rolls and rolling mill. The process is also highly flexible in terms of component and layer thickness variations.

The proposed thin metallic strips could be manufactured by different alternative techniques also. These techniques enable an appropriate or even new, mechanical properties of the strips in an easier way or at lower cost. All these procedures represent a new generation of technology and ecology-friendly procedures. One of the possible alternatives manufacturing proceeding is producing thin metallic strips using rapid solidification (melt spinning technique). If we use the classical casting it can be formed a large macro-segregation of alloying elements, grained microstructure and segregation of inter-metallic phases. This defects results in poor mechanical, corrosion and other properties of casting pre-form. In additional manufacturing phases this properties could not be improved. In many cases the properties get worse. In the case the rapid solidification conditions are totally different. The velocity of cooling is very high in the range from  $10^{1}$ - $10^{8}$  K/s and the solidification boundary are moving very fast ( $v_{sf} > 1 \text{ cm/s}$ ). Under this conditions the atoms diffusive paths becomes in liquid too short to have the thermodynamically equilibrium in the solidification boundary. Because of such in-equilibrium solidification in the microstructure becomes crystal, quasi-crystal and even amorphous-metastable phases. According to this kind of solidification the metallic strips has completely different and in some cases even excellent properties.

For the development of optimal production technology, it would be necessary to continuously monitor the influence parameters for each manufacturing phase step and immediately perform the high resolution of gained microstructure and measurements of the required metal strip's physical properties. Based on experience and previous preliminary research work, we can conclude that in the specific case of Ag and Cu metal strips, a lot of technological parameters would be necessary for monitoring. The metallic strips would be manufactured by cold rolling. The strips will enable strong and tough and also shaped in the solid state by various mechanical working processes. Their mechanical properties will be improved. A detailed knowledge of material properties changes during further thermo-mechanical treatment. Because of superficial knowledge of changes during the thermo-mechanical process, the results of simulations are significantly different from the real behaviour of the strips [5], resulting in poor physical properties. With the study and analysis of technological parameters on individual phases of the complete process of strengthening technology, especially the thermo-mechanical treatment (the temperature, atmosphere and duration of annealing, the circular velocity of cylinders, the ratio of cylinder diameter versus, step, the degree of contraction, bite angle, slitting, trimming, etc.) on the microstructure, and the final physical properties of the strips, their influence would be given exactly. Both described schematic descriptions of thermo-mechanical treatment were groundwork for assignment of Slovenian industrial patent "Production technology of Ag and Cu strips".

On the other side the aim of this research was too find possibility of producing the Cu metallic strips with demanded properties after rapid solidification (melt spinning technique), without additional thermo-mechanical treatment.

#### 2. EXPERIMENTAL WORK

Experimental work presents the production of Ag strips with dimensions (thickness – t, width - w) t × w = 0,11 mm × 1,6 mm and Cu strips with dimensions t × w = 0,11 mm × 62 mm. The melting of each component was performed in an induction furnace at T = 1375 K for Ag and at T = 1390 K for Cu under an Ar atmosphere (with Ar stream 3l/min and pressure 1.03 bar). For this purpose very high purity of components was used (about 99.99 w.%). According to the final required dimensions of strips the molten Ag or Cu were cast in a round metallic mold with recommended diameter ( $\emptyset$ ) or in a flat metallic mold with the known dimensions:

- $\phi = 16$  mm for Ag strips 0.11 mm  $\times$  1.6 mm;
- 240 mm  $\times$  200 mm  $\times$  25 mm for Cu strips 0.11 mm  $\times$  62 mm.

Ag and Cu fore-forms were cooled and then directly water quenched until the temperature was equal to the  $T_{water}$ . After quenching, the round fore-forms on the upper surface were trimmed in length to 1 cm (on the place of shrinkage). Before the heat treatment, the castings were cleaned (washing and brushed with a mixture of water and detergent) and dried. The heat treatments of Ag and Cu cast pieces were carried out in a tube furnace over various periods of time.

TM treatments were different for Ag and Cu fore-forms. Three different intermediate set of tests were done for the Ag strips the, while only two for the Cu strips. The general directions for the Ag fore-forms were:

- Profile rolling with the steps 0.5 mm up to  $\phi = 3$  mm (on a machine with the working rolls -  $\phi = 160$  mm;)

- Further profile rolling was performed on a rolling machine with smaller working rolls –  $\phi = 90$  mm with the steps 0.34 mm up to  $\phi = 1.3$  mm;

- Drawing was taken for Ag fore-forms with  $\phi = 1.3$  mm up to  $\phi = 0.85$  mm with steps 0.05 mm. After that the Ag wire was intermediate isothermally annealed at T = 853K for 10 minute in a protective atmosphere (H<sub>2</sub>:N<sub>2</sub>=80:20) and cooled slowly to room temperatures. In the second step the Ag wires with  $\phi = 0.85$  mm were again drawed up to  $\phi = 0.51$  mm with steps 0.05 mm;

- Ag wire with  $\phi = 0.51$  mm was then polish-rolled in strip with dimensions 0.11 mm × 1.6 mm with one step 0.4 mm;

- Finally the Ag strip was isothermally annealed at T = 523K for 10 minute in protective (H<sub>2</sub>:N<sub>2</sub>=80:20) according to the requested specific electrical conductivity.

The general directions for the Cu fore-forms were:

- Classical rolling of flat fore- forms with the dimensions of 240 mm  $\times$  200 mm  $\times$  25 mm up to thickness 2 mm with the steps 1 mm and at last one rolling with the step 0.2 mm;

- Cutting of Cu plate on electrical circular scissors into strips with width 62 mm (strips were then cleaned and dried);

- Classical rolling of Cu strips with width of 62 mm and thickness of 1.8 mm up to thickness 0.34 mm with step 0.365 mm. After that Cu strips were intermediate isothermally annealed at T = 933K for 30 minute in a protective atmosphere (H<sub>2</sub>:N<sub>2</sub>=80:20) and cooled slowly to room temperatures. In the second step the Cu strips with thickness of 0.34 mm were rolled again up to thickness of 0.24 mm with step 0.1 mm and one-step rolling with the step 0.06 into strips with thickness of 0.18 mm;

- After that isothermal annealing of Cu strips was performed at T = 933K for 25 minutes in a protective atmosphere (H<sub>2</sub>:N<sub>2</sub>=80:20) and slowly cooling to the  $T_{room}$ ;

- Cu strips were finally polish rolled from thickness of 0.18 mm to 0.14 mm with the step 0.04 mm and from thickness of 0.14 mm to 0.11 mm with the step 0.03 mm,

- Cu strips were trimmed with electrical circular scissors on the required strips widths.

For the rapid solidification experiments on the Faculty of Natural Sciences and Engineering Ljubljana - Chair for Engineering materials (melt spinning technique – see Figure 1), small quantities of the pure copper were prepared by induction vacuum melting using oxygen-free high-conductivity Cu as starting materials. The melts were cast into ingots 40 mm in diameter and 200 mm in height. The chemical compositions of the ingots were confirmed by wet analysis. The results show that the ingots content was 99.99% pure copper, and a small remainder represent impurities (O, C, N,). Rapidly

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solidified strips were prepared by chill-block melt spinning (wheel diameter of 350 mm) under Ar atmosphere, using different peripheral velocities of  $V_w = 10-23$  m/s. The nozzle diameter, injection overpressure, angle and the nozzle height above the Cu-Be substrate, as well as the melt superheat, were kept constant at 0.8 mm, 0.3 MPa, 6°, 8 mm and 50 K, respectively.



Figure 1: Melt spinning technique

Measurements of strip hardness were done according to standard with the Vickers test using the microhardness measurement device Zwick 3212. For testing out the samples, we used applied load F=49 N, according to standard. For every sample, we performed 12 measurements.

The electrical resistance measurements of all samples were performed by four Ni probes for electrical resistance monitoring. The electrical resistance was measured by a four probe method with a bidirectional current of 1 A, with a resolution of 1  $\mu\Omega$  and accuracy of  $\pm$  3  $\mu\Omega$ . Specific electrical conductivity (SEC) is calculated from the obtained data.

For determination of the mechanical properties the static tensile testing was performed using the tensile device Zwick/Roell ZO 10. Measurements of mechanical properties for the final state of Ag or Cu strips or wires were performed in one series with 6 wire and strip samples. The gauge length of strip was 15 mm, research conditions, as well as, shape and dimensions of tensile test strips were according to standard (the strip samples were stuck on the chucks). Measurements were performed at a constant speed of increasing deformation v = 1,5 mm/min.

Microstructural characterisation of Ag and Cu strips samples was carried out using optical microscopy (OM), scanning electron microscopy (SEM-Jeol JSM 849 A), as well as EDX microanalysis (Link analytical AN 1000).

#### **3. RESULTS**

#### Ag strips

Table 1 shows the results of hardness and specific electrical conductivity (SEC) changes of different Ag fore-forms through the TM treatment – Test 1. Marks I – VIII represent the intermediate states of Ag samples which are very important for achieving the requested final properties of Ag strips.

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Sample	Hardness [HV]	SEC [Sm/mm <sup>2</sup> ]
<b>I</b> (profile rolled, $\phi = 2.5 \text{ mm}$ )	88.6	58.3
II (annealed at $T = 853K$ for 10 minute and quenched)	40	62.4
<b>III</b> (profile rolled $\phi = 1.05 \text{ mm}$ )	92.6	57.4
<b>IV</b> (annealed at $T = 853K$ for 10 minute and quenched)	40.8	62.2
$\mathbf{V}$ (drawn, $\phi = 0.85 \text{ mm}$ )	96.3	56.3
<b>VI</b> (drawn, $\phi = 0.55$ mm)	98.7	55.1
<b>VII</b> (rolled strip, t= 0.23 mm)	89.9	55.3
<b>VIII</b> (rolled strip t= 0.16 mm)	88	55.4

Table 1: Hardness and SEC of different Ag fore-forms (samples) – Test 1

Table 2 shows the results of hardness and SEC of Test 2. The second experiment was equal to the first up to finishing the profile rolling (step 0.3 - 0.4 mm) to dimension  $\emptyset = 1.1$  mm without intermediate annealing (sample I). Afterwards the drawing of wire with the steps 0.05 mm from iz  $\emptyset = 1.1$  mm up to  $\emptyset = 0.85$  mm (sample II) and finally up to  $\emptyset = 0.513$  mm (sample III) was done. The obtained wire was rolled into strip with thickness 0.11 mm and width 1.6 mm (sample IV).

Sample	Hardness [HV]	SEC [Sm/mm <sup>2</sup> ]
Ι	92.6	58.3
II	93.43	57.9
III	95.93	57.1
IV	94.96	57.2

Table 2: Hardness and SEC of different Ag fore-forms (Test 2)

According to these results, the third production test of Ag strips was performed unchanged up to the profile rolling with the step 0.3 - 0.4 mm untill the dimension  $\emptyset = 1.3$  mm, and without any annealing. Further termo-mechanical treatment can be seen from the samples in Table 3. The average results of the mechanical properties measurement of the third set for all 6 tensile tests and results of SEC of strips are gathered and presented in Table 4.

Table 3: Hardness and SEC of different Ag fore-forms (Test 3)

Sample	Hardness [HV]	SEC [Sm/mm <sup>2</sup> ]
$\mathbf{I}$ (drawn, $\phi = 0.85$ mm)	95.8	58.2
<b>II</b> (annealed at $T = 853K$ and slowly cooled)	41.9	61.91
<b>III</b> (drawn, $\emptyset = 0.513$ mm)	109.4	60.5
<b>IV</b> (rolled strip 0,11 mm×1.6 mm)	91.5	60.7
$\mathbf{V}$ (annealed at T = 523K and slowly cooled)	71.3	61.3

The average results of mechanical testing of the Ag strips from Test 3 are shown in Table 4.

Table 4: The average value of the measurement results

	$R_m [N/mm^2]$	A [%]
Ag strip	238.04	13.51

The typically curve Stress-Strain for the final Ag strip from Test 3 is presented in Figure 2.



Figure 2: The typically stress-strain curve for final Ag strip

### Cu strips

The results of the first experimental test are collected in Table 5. Marks A0 - A1 represent the intermediate states of Cu strip-forms (samples) which are very important for achieving the requested final properties of Cu strips. Mark: A0- rolled strip with t= 0,7 mm; A1- annealed strip A0 at T=933 K in protective atmosphere (H<sub>2</sub>:N<sub>2</sub>=80:20)- 20 minute, then quenched; A2- rolled strip with t= 0,45 mm; A3- rolled strip with t= 0,285 mm; A4- rolled strip with t= 0,11 mm

Sample	Hardness [HV]	SEC [Sm/mm <sup>2</sup> ]
A0	114	55.5
A1	40	59.3
A2	101	56.5
A3	106	56.3
A4	96	57.3

Table 5: Hardness and SEC of different Cu fore-strips (Test 1)

In the second test, the initial cold deformed (sample A) and initial annealed state (sample B) of the Cu sheet with thickness 0.52 mm on the final properties of Cu strips were investigated. Annealed sheet was treated at T=933 K for 20 minute in a protective atmosphere ( $H_2:N_2=80:20$ ). Afterwards the rolling of a single sheet was taken. Table 6 shows the measured results for hardness and SEC.

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Sample	Hardness [HV]	SEC [Sm/mm <sup>2</sup> ]
A (t = $0.52 \text{ mm}$ )	94	57.5
A1 (t = 0.4 mm)	98	57.4
A2 (t = $0.25$ mm)	102	56.9
A3 (t = $0,11$ mm)	114	56.1
B (t = $0.52 \text{ mm}$ )	34	59.4
B1 (t = $0.4 \text{ mm}$ )	102	57.3
B2 (t = $0.25$ mm)	108	56.5
B3 (t = $0,11 \text{ mm}$ )	107	56.8

 Table 6. Hardness of different Cu fore-strips (Test 2)

The average results of the mechanical properties measurement for all 6 tensile tests of Cu strips are gathered and presented in Table 7.

Table 7. The average value of the measurement results

	$R_m[N/mm^2]$	A [%]
Cu strip	410.35	10.25

## Melt-spun Cu strips

Samples of the rapidly solidified strips had thickness of about 100  $\mu$ m, and two type of width: 1. 5-6 mm; 2. 2-3 mm. In the table 8 are the results of mechanical testing, hardness and SEC.

Table 8: The average value of measurement results (Rm, HV and SEC)

	$R_m [N/mm^2]$	HV 0,05	SEC [Sm/mm <sup>2</sup> ]
Melt spun Cu strip	192.62	90.15	56.9

### 4. DISCUSSION

### Ag strips

At the first experimental thermo-mechanical test of Ag strips we found out, that the hardness is very dependent on the type of mechanical treatment and increases most when the drawing (up to 50%). At the same time the specific electrical conductivity decreases for about 8%. According to obtained results of hardness and specific electrical conductivity, we could conclude that these properties are mostly influenced by the technological phase from profile rolling further; i.e. drawing, annealing and polish-rolling. On the basis of the results, we received further experimental directives: (i) the testing of productions Ag strips has to be unchanged up to finishing the profile rolling; (ii) the Ag fore-forms have to be all drawn first and then annealed in the protective atmosphere; (iii) by the drawing of Ag wire, the final diameter must be as small as possible for attaining one step polish rolling (at more steps rolling the material could softened, which is a reason for hardness fall). The results of first experiments set have namely shown that Ag strips have lower hardness, as is required. The specific electrical

conductivity is not in a gained demand, too. So the second experiment was equal to the first up to finishing the profile rolling (step 0.3 - 0.4 mm) to dimension  $\emptyset = 1.1$  mm without intermediate annealing (sample **I**). Afterwards the drawing of wire with the steps 0.05 mm from iz  $\emptyset = 1.1$  mm up to  $\emptyset = 0.85$  mm (sample **II**) and finally up to  $\emptyset = 0.513$  mm (sample **III**) was done. The obtained wire was rolled into strip with thickness 0.11 mm and width 1.6 mm (sample **IV**).

The results in the Table 2 cleary show that the final Ag strip does not correspond to the required demand within the frame work of specific electrical conductivity. According to these results, the third production test of Ag strips was performed unchanged up to the profile rolling with the step 0.3 - 0.4 mm untill the dimension  $\emptyset = 1.3$  mm, and without any annealing. Further termo-mechanical treatment can be seen from the samples in Table 3. These results show that an Ag strip produced by the decribed thermo-mechanical treatment in experimental work corresponds to the required properties: hardness and specific electrical conductivity.

With respect to the results in Table 4, we can conclude that an Ag strip fulfills all the necessary standards for mechanical properties (tensile strength  $R_m$  and elongation A).

The investigations of individual Ag fore-form stages and finally produced Ag strip microstructure have shown typically cold deformed microstructure with oriented and elongated grains. There are also many slip lines which have been formed during the cold deformation and they show the slip planes of the dislocations. Such a production method (without recrystallization annealing) was the reason for the strengthening of the material, respectively Ag strips were, consequently, reflected in the high value of hardness (>90 HV) and low value of specific electrical conductivity (55 Sm/mm<sup>2</sup>), Figure 3. Background of the microstructure formation is in the strengthening mechanism which arises from the cold deformation. When the metal is loaded in the first step, the elastic strains are attained by which the dislocations only rest. When the strains attained exceed the boundary of proportionality, the dislocations begin moving. Some dislocations could slip onto the surface and form the slip stair; others could stop in the different obstacles in the interior of the crystal. Such obstacles are: non-moving dislocations, precipitates, inclusions, grain boundary and other defects.



*Figure 3: Typically cold deformed microstructure of Ag fore-form (drawn wire)* 

#### Cu strips

Regarding the obtained results (Table 5), we can conclude that the hardness and SEC are influenced by the final manufacturing phase. The results also show that annealing lowered the hardness by approximately 50%. In this way the process must be optimally positioned in the thermo-mechanical treatment, with the optimised regime.

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The obtained measurements (Table 6) for the two Cu strips manufactured from two different initial sheets. This show that if we annealed sheet could be getting better strip properties. Also all results for SEC are within the frame work of the regulated standard. Consequently, we could conclude that the high value of hardness depends on the initial sheet state, and the final manufacturing phase.

With respect to the results in Table 7, we can conclude that Cu strip fulfills all necessary standards for mechanical properties (tensile strength  $R_m$  and elongation A).

The microstructure of Cu strips is similar to the microstructure of Ag strips. Figure 4 shows typical deformed microstructure which was formed by drawing without any annealing. The grains are elongated in the direction of rolling.



Figure 4: Microstructure of cold deformed Cu strip

Annealing of cold deformed Cu strip has caused the growth of grains, which have sizes of about 400  $\mu$ m, and different orientation (Figure 5). In this microstructure can be seen the annealing twins, which have been formed at high temperature (T = 933 K). The annealing twins have neighboured mostly the coherent borders and on a single place the non-coherent borders.



Figure 5: Microstructure of annealed Cu strips

The annealing of Cu fore-forms causes the diffusion process or recovery process. With the investigations we found out, that the size and shape of Cu grains are unchanged (the grains are fixed), while the recovery processes were run inside crystals grains. From theory it is known that point defect could be annihilated, edge dislocations climbed, opposite dislocations also annihilated, same dislocations are arranged one over another (polygonization process). The polygonization process causes small boundaries, while the crystals are divided into more sub-grains which are separated with small boundaries. Most dislocations are concentrated in small boundaries and their density in the grains is very low. These, were the reasons for the increasing Cu strips specific electrical conductivity from 50 Sm/mm<sup>2</sup> to 56 Sm/mm<sup>2</sup>, according to the decreasing of point defects, little decreasing of tensile strength (from 410 N/mm<sup>2</sup> to 370 N/mm<sup>2</sup>) and, essentially, the hardness from 100HV to 40HV.

### Melt spun Cu strips

As we expected, wheel surface velocity had a great effect on strip thickness. The relationship between strip thickness and the peripheral wheel velocity is shown in Figure 6. Decreasing the wheel velocity increases the strip thickness. In our experimental regarding melt spinning, we obtained two types of continuous strips: about 2-3 mm in width and  $< 100 \ \mu m$  in thickness; about 5-6 mm in width and  $> 100 \ \mu m$  in thickness.



Figure 6: The dependence between mean strip thickness and wheel surface velocity

On the macroscopic scale, both surfaces for all strips show the same topographic features. The free surface is always smooth and the contact surface usually exhibits some dimples, which were formed by gas picked up at the back edge of the melt puddle on the wheel's surface (Figure 7).

It is known, that at higher magnifications very fine structures with grains less than 2  $\mu$ m in size become visible on the contact surfaces of all strips (Figure 8). On the other hand, the microstructure obtained at the free surface depends on the velocity of the wheel, i.e., on the strip thickness. The surface microstructure of the thickest strips (> 100  $\mu$ m) consists of grains with relatively coarse dendritic morphology, and well-developed secondary dendrite arms. Those of the strips with medium thickness again contain at the free surface a structure with dendritic morphology, but finer in size. The thinnest strips (< 100  $\mu$ m) consist of fine grains, usually with a cellular substructure. Such microstructural changes at the free surface clearly indicate a different solidification process regarding ribbons of different thickness.



Figure 7: Topographic figure: a) lower free surface of Cu strip, b) upper free surface of Cu strip



Figure 8: Microstructure of rapidly-solidified pure Cu

Even more transparently, a solidification history was revealed in the transverse cross-sections. At medium values i.e. at strip thickness from approximately 100  $\mu$ m, the microstructure consists of three different morphological zones – fine (equiaxed), columnar and coarse equiaxed grains – which form from the bottom to the upper surface of the strip, respectively (Figure 9). With increasing velocity, the fraction of the coarse equiaxed zone decreases and the columnar zone increases. On the other hand the microstructure of the thinnest strips contains only two zones: fine equiaxed grains at the wheel side and columnar crystals at the upper side. Contrary to this, at the lowest velocity of the experiment (10 m/s), the columnar zone disappeared and the whole thickness of the strips consisted of equiaxed grains.



Figure 9: Microstructure of rapidly solidified Cu strip with 3 different morphological zones

Based on the known relationship between strip thickness and cooling rates for Cu, we estimated the mean cooling rate to be about  $10^5$  K/s for strips of thickness about > 100 µm and  $10^6$  K/s for those of thickness < 100 µm. The evolution of the microstructure depends on the solidification condition, which determines the local conditions at the solid/liquid interface. In all strips, nucleation occurred along those surfaces in contact with the quenching substrate (fine equiaxed zone). After these grains had nucleated, they grew towards the top of the casting and this resulted in the so-called columnar zone. The transition from the equiaxed to the columnar zone, as well as the grain selection, which occurs in the upper part of the fine equiaxed zone, can be understood in terms of anisotropic growth effects. Competition growth for crystals nucleated in the fine equiaxed zone into the liquid, leads to their selection. The grains which have one of their <100> crystallographic orientations most closely aligned with the heat flow direction overgrow those which have a less favourable orientation.

In a positive temperature gradient, the rate at which columnar grains grow depends on under-cooling ahead of the solidification front. In melt spinning this is affected mainly by the thickness of the melt. At an appropriate low-thickness, where the rate of growth is high enough, these grains propagate across the entire strip thickness and the columnar zone extends up to the free surface. This was obtained for strips with a thickness of about  $< 100 \,\mu\text{m}$ , cast at a wheel velocity of 23 m/s. On the other hand, if the crystals grow rate is low, nucleation ahead of the columnar grains can occur. In our experiment this happened in the upper-part of the melt during solidification of thicker strips. A columnar-to-equiaxed transition is not unusual in melt-spun strips and this is mostly a regular feature. Nucleation ahead of the columnar grains probably started by the detachment of existing dendrite arms due to convection (the melt intrusion into the mushy zone) or to thermal fluctuations. Homogeneous nucleation in the bulk liquid cannot be discounted, but this would imply a very high melt, under-cooling, in front of the dendrite tips. Newly nucleated grains can stop the growth of the columnar front, thus resulting in a columnar – equiaxed transition. The competition between the columnar and equiaxed grains is determined by the extent and degree of the undercooled liquid ahead of the columnar zone.

Finally, the results (collected in Table 8) for melt spun Cu strips show that Cu thinstrips have lower hardness, as is required, and the specific electrical conductivity and tensile strength (are also not in a gained demand). Consequently further experimentally work would focus on optimizing the production of thin Cu strips with the melt spinning technique, in regard to requested properties for strips used in the electro industry.

#### **5. CONCLUSIONS**

With the results of our examination, we can conclude that Ag and Cu strips produced in company Zlatarna Celje satisfied all the requested standards in hardness, specific electrical conductivity and mechanical properties. Hardness and SEC are mostly influenced by the technological phase from profile rolling further; i.e. drawing, annealing and polish rolling.

The investigations of individual Ag fore-form stages and, finally, the produced Ag and Cu strip microstructure, have shown typically cold deformed microstructure with oriented and elongated grains. In the microstructure there are also many slip lines which have been formed during the cold deformation. The background of this microstructure formation is in the hardening mechanism consequently, it was reflected in the high value of hardness and low value of SEC. On the other hand, the annealed microstructure has big grains and twins which have been formed during the process at high temperature and causes inversely low value of hardness and high value of SEC.

During the melt spinning experiments, we discovered that wheel surface velocity has a great effect on strip thickness. Strip thickness is, namely, the main dimension requirement in the production. The microstructure obtained at the free surface of Cu strip depends on the velocity of the wheel, i.e., on the strip's thickness.

On the other hand, the new melt spun Cu-thin strips have lower hardness, as is required, and the specific electrical conductivity and tensile strength are also not in a gained demand.

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#### **6. LITERATURE**

- [1] Liu Pei-eh and Liu Zheng: The distribution of the specific pressure in rolling strips, Applied Mathematics and Mechanics, Volume 3, Number 3 / June, 1982
- [2] Keife H., Sjogren C.: A friction model applied in the cold rolling of aluminum strips, Wear, vol. 179, nº1-2, 1994
- [3] Z.Y. Jiang A.K. Tieu, X.M. Zhang, C. Lu and W.H. Sun: Finite element simulation of cold rolling of thin strip, Available online 13 August 2003
- [4] J. S. Song, H. S. Kim, C. T. Lee and S. I. Hong: Deformation processing and mechanical properties of Cu-Cr-X (X=Ag or Co) micro-composites, Available online 15 October 2002
- [5] Sakai Y., Inoue K., Madea H.: New high-strength, high-conductivity Cu-Ag alloy sheets, Acta metall. mater., 1995