



# Effect of Severe Plastic Deformation on the Mechanical and Electrical Properties of Cr-modified Cu-Ni-Si Alloys

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## Abstract

Copper alloys are widely used in automotive, electronics, petrochemical and nuclear applications due to their good mechanical/corrosion resistance and conductivity. Although mostly beryllium containing copper alloys are utilized in these industries due to their superior properties. However, due to the toxic effects of beryllium, there is a need for alternative copper alloys. Among the alternative alloys, Cu-Ni-Si alloys known as Corson alloys are commonly preferred. Both alloying and thermomechanical treatments play important roles to attain similar or better properties in this alloy system, compared to the beryllium containing Cu alloys. In this research, a combined thermomechanical treatment is performed in order to characterize the effect of severe plastic deformation and subsequent heat treatments (i.e., precipitation hardening) on strength, hardness and electrical conductivity of Corson alloys modified by Chromium. The copper alloys having different chemical compositions (Cr contents) are processed using equal-channel angular pressing (ECAP) after solution annealing and quenching. The behavior of Cr-modified alloys is characterized in detail and the effect of the combined thermomechanical treatment and of the Cr content on the final mechanical and physical behavior is discussed. The results show that during aging of solution treated alloys containing 0.4, 0.6 and 0.8 wt-% Cr, a significant increase in both hardness and electrical conductivity is observed, and that increasing Cr content also leads to an increase of both properties. After processing by two-pass ECAP, the hardness of all investigated alloys is significantly increased compared with the solution treated state, whereas the electrical conductivity is adversely affected by severe plastic deformation. The mechanical behavior of the ECAPed alloys can be enhanced even further by performing post-ECAP aging. Therefore, it allows us to develop Cu-Ni-Si alloys by severe plastic deformation process, as an alternative to commonly used beryllium containing copper alloys. The preliminary results provide new insights for the development of construction materials that can be used in the electronic industry, high-speed rail systems, aerospace applications and nuclear power plants, which are recently high priority topics.

**Keywords:** Copper alloys, Severe plastic deformation (SPD), Equal channel angular pressing (ECAP), Mechanical and electrical properties, Precipitation (age) hardening

## Cr Modifiyeli Cu-Ni-Si Alaşımlarının Mekanik ve Elektriksel Özellikleri Üzerine Aşırı Plastik Deformasyon Etkisi

### Öz

Bakır alaşımları, sahip oldukları yüksek mukavemet, korozyon direnci ve iletkenlikleri nedeniyle otomotiv, elektronik, petrokimya ve nükleer uygulamalarında yaygın olarak kullanılmaktadır. Bu endüstrilerde üstün özelliklerinden dolayı çoğunlukla berilyum içeren bakır alaşımları kullanılmakla birlikte, berilyumun toksik etkileri nedeniyle alternatif bakır alaşımlarına ihtiyaç duyulmaktadır. Bunlar arasında *Corson* alaşımları olarak bilinen Cu-Ni-Si alaşımları yaygın olarak tercih edilmektedir. Hem alaşımlama hem de termomekanik işlemler, bu alaşım sisteminde berilyum içeren Cu alaşımlarına kıyasla benzer ve hatta daha iyi özellikler elde etme

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noktasında önemli rol oynamaktadır. Bu araştırmada, aşırı plastik deformasyon ve akabinde gerçekleştirilen ısıl işlemlerin (çökeltme sertleşmesi) Cr ile modifiye edilmiş Cu-Ni-Si alaşımlarının mukavemeti, sertliği ve elektrik iletkenliği üzerine etkisini karakterize etmek için kombine termomekanik işlem uygulanmıştır. Farklı kimyasal bileşime (Cr içeriğine) sahip bakır alaşımları, çözeltiliye alma ve su verme işleminden sonra eş kanallı açılal pres (EKAP) yöntemi kullanılarak şekillendirilmiştir. Cr modifiyeli alaşımların davranışı ayrıntılı olarak karakterize edilmiş ve kombine termomekanik işlem ile alaşımdaki Cr içeriğinin nihai mekanik ve fiziksel özellikler üzerine etkisi irdelenmiştir. Elde edilen sonuçlar, ağırlıkça %0,4, 0,6 ve 0,8 Cr içeren çözeltiliye alınmış alaşımların yaşlandırılması sırasında hem sertlikte hem de elektriksel iletkenlikte önemli bir artış gözlemlendiğini ve artan Cr içeriğinin Cu-Ni-Si alaşımlarının sertliğini ve iletkenliğini artırdığını göstermektedir. İki paso EKAP ile işlemi neticesinde, incelenen tüm alaşımların sertliği çözeltiliye alınmış duruma kıyasla önemli ölçüde artarken, elektriksel iletkenliği aşırı plastik deformasyondan negatif etkilenmiştir. EKAPlanmış alaşımlarının mekanik özellikleri, EKAP sonrası yaşlandırma ile daha da geliştirilebilmektedir. Bu nedenle, yaygın olarak kullanılan bakır-berilyum alaşımlarına alternatif Cu-Ni-Si alaşımlarının aşırı plastik deformasyon ile geliştirilmesi mümkündür. İlk sonuçlar, son zamanların yüksek öncelikli alanları arasında yer alan elektronik endüstrisi, yüksek hız raylı sistemler, uzay/havacılık uygulamaları ile nükleer santrallerde kullanılabilecek yapı malzemelerinin geliştirilmesine ışık tutmaktadır.

**Anahtar Kelimeler:** Bakır alaşımları, Aşırı plastik deformasyon, Eş kanallı açılal pres, Mekanik ve elektriksel özellikler, Çökeltme sertleşmesi

## 1. Introduction

Copper and its alloys are used in many different industries such as electronics, automotive and nuclear industries. The range of applications of Cu alloys is extended due to their good strength, wear and corrosion resistance, and high electrical conductivity (Lei et al., 2013a; Lockyer and Noble, 1994). Most of these alloys are generally precipitation hardened and are alloyed with alloying elements (with very low solubility) such as Cr to maintain high electrical conductivity. Cu-Be, Cu-Co-Ni-Be, Cu-Cr-Zr and Cu-Ni-Si alloy families belong to the group of precipitation hardenable copper alloys (Monzen and Watanabe, 2008; Woodcraft et al., 2001; Xie et al., 2009). Various studies on these alloys have been performed to optimize hardness and conductivity as a function of different alloying and heat treatment processes (Xiao, 2020). Although Be containing Cu alloys with their high strength and conductivity are among the precipitation hardenable copper alloys, the toxicity of beryllium and its low stress relaxation at high temperatures require the development of new alloy systems instead of these alloys (Lei et al., 2013a; Lei et al., 2013b).

Zhao et al. (2003) characterized the aging behavior of Cu-Ni-Si alloys. Furthermore, Watanabe et al. (2018) and Li et al. (2009) investigated the deformation behavior of a Cu-Ni-Si alloy processed by high-pressure torsion (HPT) and the microstructural evolution of alloys modified by Sn and Mg, respectively. The microstructure and properties of a high-strength Cu-Ni-Si alloy developed with addition of Co and Cr were analyzed by Krishna et al. (2013). They have reported that aging of the solution treated samples leads to a significant increase in strength and electrical conductivity which is mainly attributed to the formation of precipitates. The contribution of thermomechanical processes, alloying and Ni:Si ratio to the improvement of the alloy properties is remarkable. The hardness values of the modified Cu-Ni-Si alloys processed by conventional thermomechanical treatments (rolling, forging, etc.) with subsequent aging are given in Figure 1a. Cu-Ni-Si alloys can reach hardness values ranging from 190 to 350 HV as a function of both composition and thermomechanical processes. Moreover, high hardness values can only be achieved with high Ni:Si ratios and alloying. The modification and thermomechanical processes would lead to superior mechanical behavior of Cu-Ni-Si alloys compared to Be containing Cu alloys. The change in electrical conductivity levels of these alloys is given in Figure 1b. Although high Ni:Si ratios provide high strength of the alloys, the electrical conductivity can be

significantly reduced with increasing Ni:Si ratio. However, the electrical conductivity values (25-50 IACS %) of the modified and thermomechanically treated Cu-Ni-Si alloys are higher than the conductivity value of the Cu-Be alloys (22 IACS %). Considering both Figures 1a and b, it becomes clear that there is a need for new processes to further enhance the mechanical properties of copper alloys, although it is seen that modified alloys exhibit superior mechanical properties if conventional thermomechanical processes are diversified.

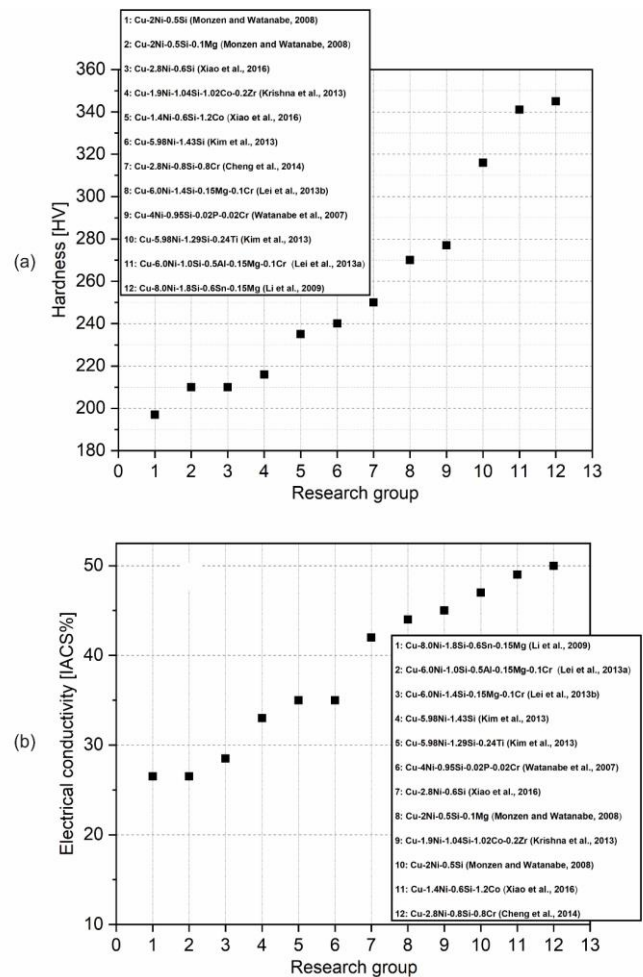


Figure 1. (a) Hardness and (b) electrical conductivity values of modified/processed (by conventional thermomechanical treatments) Cu-Ni-Si alloys investigated by different research groups

Various severe plastic deformation (SPD) methods, particularly equal-channel angular pressing (ECAP) and HPT with subsequent aging processes are used to enhance the mechanical and physical behavior of the alloys due to the grain refinement during SPD process and due to the precipitation hardening during subsequent aging (Valiev et al., 2002; Valiev and Langdon, 2006). Among the SPD techniques, ECAP is widely used to produce ultrafine-grained materials having excellent properties (Valiev and Langdon, 2006; Furukawa et al., 2001; Furuno et al., 2004). Khereddine et al. (2013) have been working on the ECAP processing of Cu-Ni-Si alloys. They processed Cu-2.5Ni-0.6Si alloys with a constant Ni:Si ratio of 5:1 by ECAP and HPT and reported on the change of mechanical properties after SPD. They concluded that the hardness increases with increasing number of torsion revolutions and reaches the highest value of 285 HV after 10 revolutions, whereas the mean grain size and dislocation density of the alloy are equal to 38 nm and  $7.59 \times 10^{15} \text{ m}^{-2}$ , respectively. In the alloy processed by ECAP after 12 passes, the highest hardness value (195 HV) is reached and the mean grain size and the dislocation density are equal to 44 nm and  $4.91 \times 10^{15} \text{ m}^{-2}$ , respectively. As can be clearly seen, ultrafine-grained structures and increasing the dislocation density in the matrix achieved by SPD processes provide a significant improvement of the properties of this alloy system. In this work, the effect of ECAP process and precipitation hardening during subsequent aging as well as of the Cr addition/content on the mechanical and physical behavior of Cu-Ni-Si alloys having different Cr contents is investigated systematically.

## 2. Material and Method

In this work, the effect of ECAP process and precipitation hardening on the mechanical and electrical properties of Cu-Ni-Si alloys modified by Cr was investigated. The chemical compositions of the investigated alloys are shown in Table 1. Ni:Si ratio in all investigated alloys is constant (~2:1) and the content of Cr alloying element is equal to 0.4, 0.6 and 0.8.

Table 1. Chemical compositions of the Cu alloys investigated in this study

Alloy	Cu	Ni	Si	Cr
CuNiSi-Cr	98.2	0.25	0.107	0.4
CuNiSi-Cr	99	0.035	0.015	0.6
CuNiSi-Cr	98.8	0.04	0.026	0.82

The solution annealing with subsequent quenching and aging, ECAP processing and post-ECAP aging at 450 °C for 2, 4, 6 and 8 h were performed (s. Figure 2) and after each step, the mechanical and electrical properties were characterized by the quasi-static, uniaxial tensile tests ( $\dot{\epsilon} \sim 10^{-3} \text{ s}^{-1}$ ), the hardness and electrical conductivity measurements with regard to the effect of the combined thermomechanical treatment (i.e., ECAP + post-ECAP aging / precipitation hardening) and the Cr content on the alloy properties.

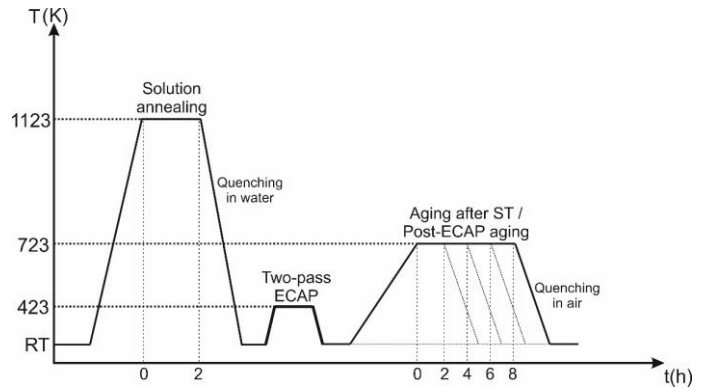


Figure 2. Procedure of the conventional heat treatment vs. combined thermomechanical treatment of the investigated Cu alloys

Two-pass ECAP processing of the investigated Cu alloys were performed at 423 K using a die with a channel/corner angle of  $90^\circ/0^\circ$  via route Bc ( $90^\circ$  counterclockwise rotation between passes) (s. Figure 3). During ECAP processing, the pressing speed of hydraulic press with 120 tons capacity was set to  $20 \text{ mm} \cdot \text{min}^{-1}$ .

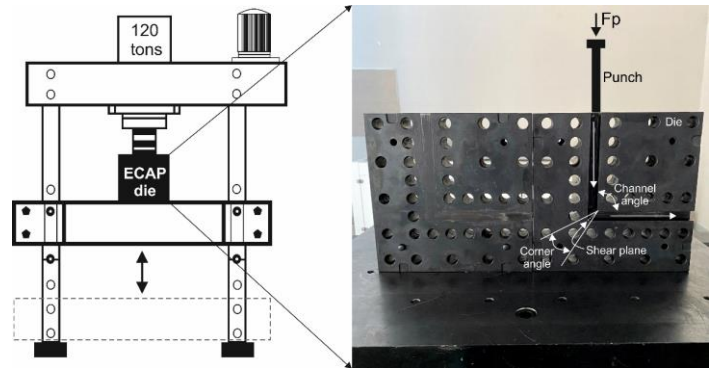


Figure 3. Hydraulic press and ECAP die with the channel angle of  $90^\circ$  and corner angle of  $0^\circ$  used in this study

Tensile tests were performed using a universal testing machine (Schimadzu AGX-V 100 kN) at room temperature under quasi-static loading conditions ( $\dot{\epsilon} \sim 10^{-3} \text{ s}^{-1}$ ). The microhardness measurements (with a load of 0.5 kg and dwell time of 20 s) were conducted using a Vickers microhardness tester (Microbul 1000 DN-AT). Electrical conductivity of Cu alloys was measured using an eddy current electrical conductivity meter (AutoSigma 3000DL). Samples for tensile tests, hardness and electrical conductivity measurements were cut from the center of the non-ECAPed and ECAPed billets along the longitudinal (ECAP) direction (s. Figure 4).

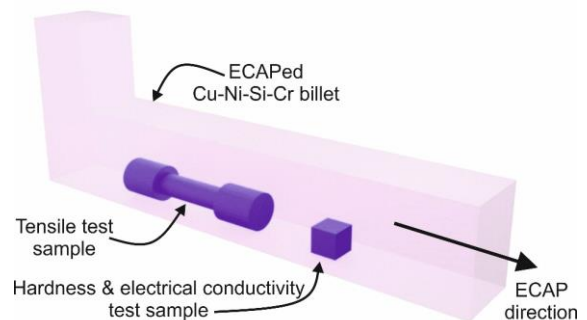


Figure 4. Schematic illustration of the cutting direction of samples for the characterization

### 3. Results and Discussion

In Figure 5a and b, the measured hardness and electrical conductivity values of the Cu-Ni-Si-Cr alloys processed by conventional heat treatment (solution treatment (ST) + aging) and by combined thermomechanical treatment (ST + ECAP + post-ECAP aging) are plotted as a function of aging time and Cr content. During aging at 723 K, the hardness of both ST and ECAPed alloys increase with increasing aging time due to precipitation with a homogeneous distribution (Cheng et al., 2014). The precipitation formation leads to decreasing solute content in the matrix and decreasing electrical resistivity (reciprocal of electrical conductivity) (Lei et al., 2017; Ahn et al., 2020). Therefore, increasing aging time (i.e., increasing volume fraction of precipitates) results in increasing conductivity in all investigated state. In early phase of aging, the increase in conductivity is more pronounced due to the faster precipitation. Furthermore, both the hardness and conductivity of the investigated alloys increase with increasing Cr content. The highest value of the electrical conductivity (~50 IACS%) is reached in the alloy with 0.8% Cr after processing by conventional heat treatment. ECAP processing results in decreasing electrical conductivity of all investigated alloy groups with different Cr contents due to the increasing resistivity (increased electrons scattering) by severe plastic deformation (Arenas et al., 2015), whereas the hardness of ECAPed alloys is significantly higher compared with ST alloys due to the increased number of grain boundaries of the material processed by ECAP. The hardness and electrical conductivity values measured as a function of different investigated states and Cr contents are listed in Table 2.

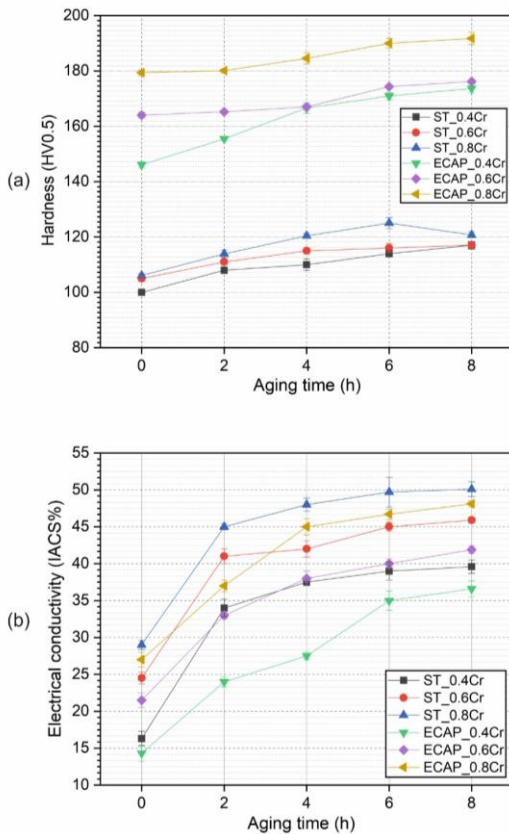


Table 2. Hardness and electrical conductivity values of Cu-Ni-Si-Cr alloys for different states and Cr contents investigated in this study

Investigated state	Cr content (%)	ECAPed + Peak aged (6 h)	ST + Peak aged (6 h)	ST
Hardness (HV0.5)	0.8	190	126	108
	0.6	174	116	106
	0.4	170	114	100
Electrical conductivity (IACS%)	0.8	46.5	50	27.5
	0.6	40	45	24
	0.4	35	39	16

Figure 6 shows the engineering stress-strain curves for solution treated (ST), peak aged (PA at 723 K for 6 h) and ECAPed samples with different Cr contents under uniaxial tensile loading at room temperature and a  $\dot{\epsilon} \sim 10^{-3} \text{ s}^{-1}$ . The ultimate strains and tensile strengths obtained from mechanical responses are listed in Table 3. Two-pass 90° ECAP processing results in a considerable increase of tensile strength of all investigated alloys compared to the ST samples due to the grain refinement by ECAP according to the *Hall-Petch relation* (Petch, 1953) and due to the increasing dislocation density by strain hardening. Cu-Ni-Si alloy with 0.8% Cr exhibits the highest tensile strength of ~360 MPa, ~570 MPa and ~470 MPa in ST, PA and ECAPed states, respectively. After processing by two-pass ECAP, only one tensile test specimen could be cut from the ECAPed billet. However, it is well-known that the post-ECAP peak aging should lead to a higher tensile strength than that of 570 MPa in PA state due to the subsequent additional precipitation hardening (as can also be seen in the hardness plots in Figure 5a based on the general relation between strength and hardness). In multi-pass ECAPed alloys, it is expected that plastic deformation mainly occurs by twinning instead of dislocation slip due to the high dislocation density which strongly hinders the dislocation motion. The twinning mechanism has a similar effect on the strengthening of the materials as the grain refinement. ECAP processing results in higher tensile strengths of Cu-Ni-Si alloys with 0.6% and 0.4% Cr compared with that after solution treatment followed by peak aging. The mechanical properties can be improved even further by post-ECAP aging.

Figure 5. (a) Hardness and (b) electrical conductivity values of the solution treated (ST) and ECAPed Cu-Ni-Si alloys modified by 0.4, 0.6 and 0.8% Cr as a function of aging time at 723 K

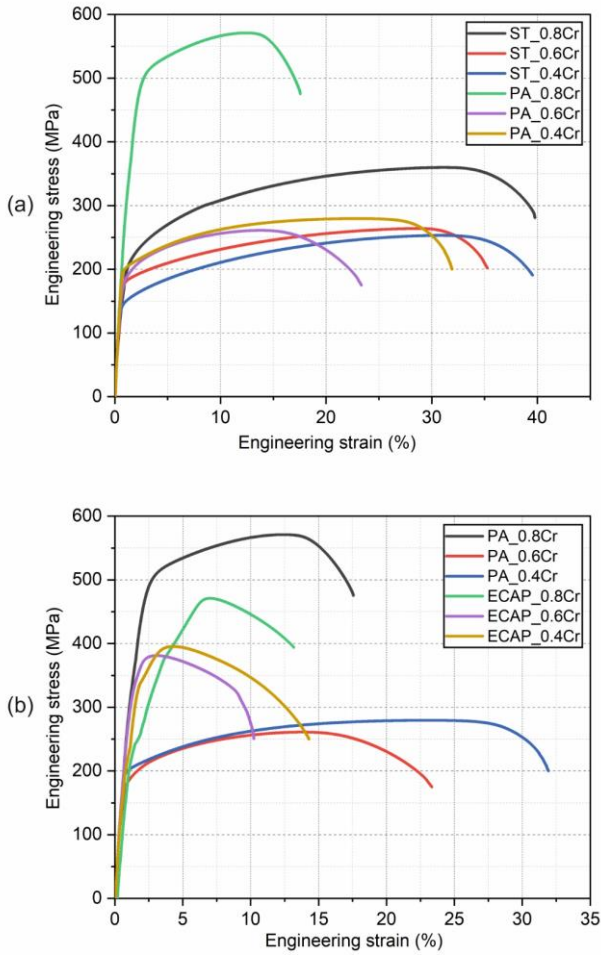


Figure 6. Engineering stress-strain curves of Cu-Ni-Si-Cr alloys with different Cr contents for (a) ST and PA states and (b) PA and ECAPed state

It is noteworthy that the ultimate strain (i.e., the ductility) and the uniform elongation of the Cu-Ni-Si-Cr alloys are strongly decreased by ECAP processing (s. Table 3). This is attributed to the early-stage strain (deformation) localization and subsequent early-stage fracture that may occur in fine grained metals processed by severe plastic deformation due to the prevention of the formation and accumulation of new dislocations by means of the microstructural characteristics such as high/critical dislocation density, substructures formed in elongated grains, and high internal stresses (Frint and Wagner, 2004; Ma et al., 2009).

#### 4. Conclusions and Recommendations

In this study, the effect of ECAP and Cr content on the mechanical and electrical properties of precipitation hardening Cu-Ni-Si alloys was systematically characterized by subjecting the alloys to both the conventional heat treatment and the combined thermomechanical treatment. The results are summarized below:

- The hardness of Cu-Ni-Si-Cr alloys increases with increasing Cr content (in the investigated range of 0.4 to 0.8): After solution treatment, the hardness values of the alloys with 0.8% Cr, 0.6% Cr and 0.4% Cr content are equal to 108, 106 and 100 HV0.5, respectively. Furthermore, two-pass ECAP processing results in a significant increase of

hardness for all investigated alloy compositions: After aging of the alloys with 0.8% Cr, 0.6% Cr and 0.4% Cr content at 723 K for 6 h, an increase of ~51%, ~50% and ~49% occurs, respectively. This is attributed to the mechanisms of strain hardening and grain refinement by ECAP processing, and of precipitation hardening by aging.

- An increase in the Cr content leads to increasing conductivity of the Cu-Ni-Si-Cr alloys, whereas ECAP processing results in a significant decrease of electrical conductivity (i.e., increase of resistivity) due to the increased probability of electrons scattering by severe plastic deformation. The conductivity increases during aging at 723 K with increasing aging time due to the decreasing solute content in the matrix caused by the formation of precipitates.
- After processing of the Cu alloys investigated in this study by ECAP, due to the strain hardening and grain refinement, the yield (and tensile) strength is increased significantly, whereas the ductility of the ECAPed alloys is decreased considerably due to the localization of the deformation during the ECAP process, which may lead to an early-stage strain localization during post-ECAP plastic deformation.

Further investigations on Cu-Ni-Si alloys with different Ni:Si ratios and Cr contents should be performed by ECAP processing, and new strategies should be developed to solve the problems that may occur in the processing of modified alloys, especially in the case of high Ni:Si ratios. Thus, a matrix of Cu-Ni-Si alloys that exhibits high strength and electrical conductivity after severe plastic deformation can be designed with varying the Ni:Si ratio and content of the alloying elements.

#### 5. Acknowledge

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Table 3. Ultimate strain and tensile strength values of Cu-Ni-Si-Cr alloys for different states and Cr contents investigated in this study

Investigated state	Solution treated (ST)			Peak aged (PA)			ECAPed			
	Cr content (%)	0.4	0.6	0.8	0.4	0.6	0.8	0.4	0.6	0.8
Tensile strength (MPa)		250	255	360	270	265	570	390	383	470
Ultimate strain (%)		39	35	40	32	23	17.5	14	9	14

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