Structure and properties of CuFe2 alloy

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ABSTRACT

Purpose: The objective of this work was to investigate the changes taking place in the structure and properties of CuFe2 alloy caused by combined heat treatment and metal working. The objective of this paper was to describe phenomena related to the formation of functional properties CuFe2 strips, especially for obtaining hardness in 120-140 HV range and electrical conductivity above 35 MS/m.

Design/methodology/approach: The investigated material consisted of two industrial melts of CuFe2. Systematic investigations of selected variants of heat treatment and plastic working operations were carried out. The investigations started with description of microstructure and properties in initial state, after quenching, after cold working, quenching and ageing, after quenching and ageing, after quenching and cold working and after cold working and annealing - omitting quenching and ageing process. Hardness test (HV) and electrical conductivity were determined on strip samples. Typical tension tests and metallographic investigations were also carried out.

Findings: Structure and properties of industrial CuFe2 alloy differs significantly from the literature descriptions, especially after quenching process. It could be assumed, that the dissolved in a melting process alloy additives (in this case a part of dissolved iron) might be supersaturated, but some of them might be precipitated. This theory was confirmed by the results of investigation into mechanical properties, microstructure and electrical conductivity.

Practical implications: The presented investigation results, besides their cognitive values, provide many useful information which might be implemented in an industrial practice.

Originality/value: It was assumed that cold deformation with rolling reduction 70% and annealing at temperature 480°C for 12 hours provided possibilities to reach maximal electrical conductivity 37 MS/m and maximal hardness 136 HV.

Keywords: Metallic alloys; Functional materials; Metallography; Heat treatment

Reference to this paper should be given in the following way:

1. Introduction

A lot of attention was given to investigation of CuFe2 alloy in sublime laboratory conditions both in the world [1-3] and Polish [4-11] publications. These investigations were focused on production of charging material (melting and casting in a vacuum furnace), high temperature homogenizing in hydrogen atmosphere for about fifteen hours, holding before precipitation in salt baths or in glass capsules, subtle investigations by optical microscopy, scanning, transmission or high resolution electron microscopy, X-ray or magnetic investigations and others. It should be notified that the investigations were conducted on low mass samples [12-14].

The presented research results explained the phenomena which take place in alloys structure related to precipitation and ageing also with cold deformation, especially during martensite
transformation of precipitated iron particles in ageing process. However, they contain almost no data combining heat treatment and metal working parameters with structure shaping and obtaining of intended functional properties (also from industrial point of view) of CuFe2 alloy strips. The objective of this paper was to describe phenomena related to the formation of functional properties of CuFe2 strips, especially for obtaining hardness in 120-140 HV range and electrical conductivity above 35 MS/m.

2. Technical requirements for CuFe2 strips

The standards or technical requirements specify basic functional properties: chemical composition, mechanical properties, dimension ranges with tolerances, electrical and thermal conductivity, surface quality, corrosion resistance and others. Sample requirements were presented in Tables 1-4.

3. Investigated material

The investigated material consisted of two industrial melts of CuFe2 alloy rolled from thickness 15 mm to 4 mm or to 2 mm, directly after continuous casting and surface machining. The material was then annealed at temperature of 520°C (heating to the 520°C for 3.5 hours, holding in 520°C for 5 hours, cooling to the room temperature for 8 hours). Hardness test (HV) and electrical conductivity were determined on strip samples of thickness 4.0 and 2.0 mm. Typical tension tests and metallographic investigations were also carried out. Chemical composition of continuous cast alloys are presented in Tables 5 and 6. Investigation results of hardness and electrical conductivity in initial samples are presented in Table 7. Investigation results of mechanical properties in initial samples are presented in Table 8.

Strip microstructure after rolling from thickness 15 mm to 4.0 mm and 2.0 mm was highly deformed and presented characteristic bands (Figs. 1 and 2).

<table>
<thead>
<tr>
<th>Material Mark</th>
<th>State</th>
<th>Nominal thickness [mm]</th>
<th>UTS [MPa]</th>
<th>Elongation A50 [%]</th>
<th>Hardness HV5</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuFe2P</td>
<td></td>
<td>from</td>
<td>to</td>
<td>min</td>
<td>max</td>
</tr>
<tr>
<td>R370</td>
<td></td>
<td>0.10</td>
<td>2.0</td>
<td>370</td>
<td>430</td>
</tr>
<tr>
<td>H120</td>
<td></td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>R420</td>
<td></td>
<td>0.10</td>
<td>2.0</td>
<td>420</td>
<td>480</td>
</tr>
<tr>
<td>H130</td>
<td></td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>R470</td>
<td></td>
<td>0.10</td>
<td>1.0</td>
<td>470</td>
<td>530</td>
</tr>
<tr>
<td>H140</td>
<td></td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>R520</td>
<td></td>
<td>0.10</td>
<td>1.0</td>
<td>520</td>
<td>580</td>
</tr>
<tr>
<td>H150</td>
<td></td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 1. Chemical composition of CuFe2 alloy (PN-EN 1758 “Copper and copper alloys. Strips for lead frames”)

<table>
<thead>
<tr>
<th>Cu</th>
<th>Fe</th>
<th>Zn</th>
<th>P</th>
<th>Pb</th>
<th>Sn</th>
<th>Ni</th>
<th>Others</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rest</td>
<td>2.1-2.6</td>
<td>0.05-0.20</td>
<td>0.053</td>
<td>0.015-0.15</td>
<td>-</td>
<td>-</td>
<td>Max. 0.2</td>
</tr>
</tbody>
</table>

Table 2. Mechanical properties of CuFe2 alloy (PN-EN 1758 “Copper and copper alloys. Strips for lead frames”)

Fig. 1. Microstructure of CuFe2 alloy in initial state. Microsection perpendicular to the rolling direction, thickness 4.0 mm

Fig. 2. Microstructure of CuFe2 alloy in initial state. Microsection perpendicular to the rolling direction, thickness 2.0 mm
### 4. Investigation results

To obtain the intended hardness in range 120-140 HV and electrical conductivity above 35 MS/m, systematic investigations of selected variants of heat treatment and plastic working operations were carried out. The investigations started with description of microstructure and properties in initial state, after quenching, after cold working, quenching and ageing, after quenching and ageing, after quenching, ageing and cold working and after cold working and annealing - omitting quenching and ageing process.

#### 4.1. Quenching

Hardness and electrical conductivity investigations were used for evaluation of CuFe2 alloy quenching process. Expensive and labour–consuming tension test were abandoned. 2.0 mm thickness samples (oxidation protected) were held at temperature of 800°C, 900°C and 1000°C for 1 and 3 hours, then quenched in water. Investigation results of hardness and electrical conductivity after precipitation are presented in Table 9.
Table 9. Investigation results of hardness and electrical conductivity after quenching of CuFe2 alloy

<table>
<thead>
<tr>
<th>Quenching parameters</th>
<th>Investigation results of hardness [HV5] and electrical conductivity [MS/m]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temp. [°C]</td>
<td>Time [hours]</td>
</tr>
<tr>
<td>800</td>
<td>1</td>
</tr>
<tr>
<td>800</td>
<td>3</td>
</tr>
<tr>
<td>900</td>
<td>1</td>
</tr>
<tr>
<td>900</td>
<td>3</td>
</tr>
<tr>
<td>1000</td>
<td>1</td>
</tr>
<tr>
<td>1000</td>
<td>3</td>
</tr>
</tbody>
</table>

where:

HV5 – Vickers hardness
sHV – Vickers hardness standard deviation
n - number of measurements
γ – electrical conductivity
sγ – electrical conductivity standard deviation

The examinations of electrical conductivity after different heat treatment processes are widely performed for copper alloys, because they are easy and inexpensive when compared to the X-ray investigations. Data collected in Table 9 show that quenching process parameters of CuFe alloy has a strong influence on changes of hardness and electrical conductivity. With increase of quenching temperature hardness also increases, but electrical conductivity decreases. Similarly, significant changes in microstructure were observed (Figs. 3 and 4).

Quenching at temperature of 800°C (held for 1 hour) did not cause grains growth. Grains size started to change from quenching temperature of 900°C, and after holding at temperature of 1000°C clear grains growth and formation of twins boundaries were observed. Interesting results (SEM) were obtained for CuFe2 samples quenched at temperature 1000°C held for 1 and 3 hours. Undissolved iron particles of different size became visible (Fig. 5).

Fig. 3. Microstructure of CuFe2 alloy after quenching at temperature 800°C, held for 1 hour, optical microscopy

Fig. 4. Microstructure of CuFe2 alloy after quenching at temperature 1000°C, held for 1 hour, optical microscopy

Fig. 5. Microstructure of CuFe alloy after quenching at 1000°C for 1 hour, SEM
The microstructure shown on Fig. 6 (held for 3 hours) contained no small iron particles, these particles were probably dissolved in copper matrix.

![Fig. 6. Microstructure of CuFe alloy after quenching at 1000°C for 3 hour, SEM](image)

Hardness and electrical conductivity of CuFe alloy quenched at temperature of 800°C changed significantly. Detailed investigations of structure changes were carried out by transmission electron microscopy. Uniformly distributed precipitated iron particles of globular shape were observed (Fig. 7a). Smaller iron particles were seen over the dislocations (Fig. 7b). For confirmation of presence of iron particles in a structure of quenched CuFe2 alloy, electron diffraction and its solution are presented in Fig. 8. EBSD method was used for evaluation of crystallographic relations of individual structure elements. This method showed fibrous structure (direction (111)) in the sample quenched at temperature of 800°C. This orientation dominated in the structure, about 75% of grain were oriented in this direction.

![Fig. 8. Electron diffraction of precipitated γ-Fe particle.](image)

### 4.2. Cold working, quenching and ageing

Initial material was cold worked with rolling reduction 20% (4.0 mm-3.18 mm), 30% (4.0 mm-2.70 mm), 50% (4.0 mm-1.94 mm) and 70% (4.0 mm-1.22 mm), and then mechanical properties and electrical conductivity were investigated. Research results are presented in Table 10. The results of mechanical properties determinations are presented graphically in Fig. 9.

It was discovered, that hardness did not change significantly up to 70% of rolling reduction, and for this rolling reduction the lowest electrical conductivity was obtained. In Fig. 8 average values of ultimate tensile strength (UTS), yield strength (YS) and elongation (A10) for initial samples (after cold rolling from 15mm to 4.0 mm and annealing) are marked on γ-axis. Average UTS value was 434 MPa, average YS value was 373 MPa and average elongation A10 value was 10.8%.
Table 10.
Research results of hardness and electrical conductivity versus rolling reduction of CuFe2 alloy

<table>
<thead>
<tr>
<th>Properties</th>
<th>Number of measurements</th>
<th>Research results of hardness [HV5] and electrical conductivity [MS/m.]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average hardness HV₅</td>
<td>100</td>
<td>153.7  154.4  155.4  166.6</td>
</tr>
<tr>
<td>Hardness standard deviation HV</td>
<td>100</td>
<td>0.56  0.74  0.64  0.71</td>
</tr>
<tr>
<td>Average electrical conductivity [MS/m]</td>
<td>100</td>
<td>24.65  22.63  20.76  18.08</td>
</tr>
<tr>
<td>Electrical conductivity standard deviation [MS/m]</td>
<td>100</td>
<td>0.42  0.47  0.51  0.53</td>
</tr>
</tbody>
</table>

Fig. 9. Rolling reduction effect on ultimate tensile strength (UTS), yield strength (YS) and elongation (A₁₀) of CuFe2 alloy

Samples deformed with rolling reduction: 20%, 30%, 50%, and 70% were held at 1000°C for 1 hour then quenched in water. Investigations into hardness, electrical conductivity and mechanical properties were carried out. Results are shown in Table 11 and in Fig. 10.

Table 11.
Hardness and electrical conductivity of CuFe2 alloy after deformation and quenching process (1000°C/1hour/water)

<table>
<thead>
<tr>
<th>Rolling reduction [%]</th>
<th>Number of measurements</th>
<th>Investigation results of hardness and electrical conductivity after deformation and quenching process (1000°C/1hour/water)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>20</td>
<td>HV₅  sHV  χ  sχ</td>
</tr>
<tr>
<td>30</td>
<td>20</td>
<td>66.1  0.98  14.35  0.44</td>
</tr>
<tr>
<td>50</td>
<td>20</td>
<td>65.3  0.95  14.18  0.53</td>
</tr>
<tr>
<td>70</td>
<td>20</td>
<td>64.9  0.97  14.07  0.57</td>
</tr>
</tbody>
</table>

where:

HV₅ – Vickers hardness
sHV – Vickers hardness standard deviation
n – number of measurements
χ – electrical conductivity
sχ – electrical conductivity standard deviation

Tension test were carried out with samples deformed (with different rolling reduction) and quenched. Results are presented in Fig. 10. The values of ultimate tensile strength (UTS), yield strength (YS) and elongation (A₁₀) for samples after quenching process only (initial samples, 4.0mm thickness) are marked on y-axis.

Fig. 10. Rolling reduction effect on ultimate tensile strength (UTS), yield strength (YS) and elongation (A₁₀) of CuFe2 alloy after cold rolling and quenching process (1000°C/1hour/water)

Cold deformed (with different rolling reduction) and quenched samples were aged at temperature of 450°C, 500°C, 550°C, 600°C for 5, 30, 60, 120, 420 minutes. Hardness and electrical conductivity changes versus ageing time at temperature of 500°C (in which the highest hardness and electrical conductivity values were obtained) are presented in Fig. 11 and Fig. 12.

Fig. 11. Hardness changes versus ageing time at temperature 500°C for quenched CuFe2 alloy
Materials

Structure and properties of CuFe2 alloy

Microstructure of CuFe2 alloy examined by TEM (deformed with rolling reduction 20%, quenched (1000°C/1 hour/water) and aged at temperature of 550°C for 120 minutes) contains precipitated, coherent iron particles (Fig. 13 and Fig. 14), precipitation-free grain boundaries area and primary iron particles (Fig. 15).

Fig. 12. Electrical conductivity changes versus ageing time at temperature 500°C for quenched CuFe2 alloy

Fig. 13. Microstructure of CuFe2 alloy, cold deformed with rolling reduction 20%, quenched (1000°C/1 hour/water) and aged at temperature of 550°C for 120 minutes. TEM

The investigated processing method which covered cold deformation before quenching and ageing did not result in intended values of hardness and electrical conductivity. Hardness of CuFe2 alloy after processing was below 100 HV and electrical conductivity was in the range 30-35 MS/m.

4.3. Quenching and ageing

Quenched samples of thickness 4.0 mm and 2.0 mm were aged at temperature of 480°C for 1, 2, 4, 6, 10, 12, and 15 hours. Changes of hardness and electrical conductivity versus ageing parameters are presented in Fig. 16 and Fig. 17, and represent the average value of 6 measurements.

Hardness of 2.0 mm samples increased insignificantly during ageing from 69 HV (after quenching process) to 91 HV (after 1 hour of ageing) and to 95 HV (after 15 hours of ageing). Electrical conductivity increased from 12 MS/m (after quenching process) to 30.7 MS/m (after 1 hour of ageing) and to 36.5 MS/m (after 12 hours of ageing). Hardness changes of 4.0 mm samples were more significant. It increased from 56 HV (after quenching process) to 95 HV (after 1 hour of ageing) and to 106 HV (after 6 and 10 hours of ageing). Hardness decreased to the 100 HV after 15 hours of ageing. Electrical conductivity increased from 12 MS/m (after quenching process) to 30 MS/m (after 1 hour of ageing). Maximal value of electrical conductivity 35.3MS/m was obtained after 6 hours of ageing.
Investigated processing method did not result in the intended values of properties (120-140HV, above 35 MS/m).

### 4.4. Quenching, ageing and cold deformation

In this processing method strips of CuFe2 alloys quenched at 1000°C and aged at 480°C for 6 hours were cold rolled.

The highest electrical conductivity 32 MS/m was obtained for rolling reduction 10%. Research results are presented in Fig. 18. The intended hardness values (120-140 HV) were obtained for rolling reduction 10% and 20% (Table 12). However, the electrical conductivity in this conditions was 32.5 MS/m and 31.5 MS/m, respectively, which means that the intended set of properties was not reached.

### Table 12.

<table>
<thead>
<tr>
<th>Sample</th>
<th>UTS MPa</th>
<th>YS MPa</th>
<th>Elongation A %</th>
<th>HV</th>
<th>γ MS/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>320</td>
<td>155</td>
<td>35</td>
<td>65</td>
<td>14</td>
</tr>
<tr>
<td>2</td>
<td>400</td>
<td>360</td>
<td>12</td>
<td>97</td>
<td>33.4</td>
</tr>
<tr>
<td>3</td>
<td>410</td>
<td>380</td>
<td>10</td>
<td>136</td>
<td>32</td>
</tr>
<tr>
<td>4</td>
<td>440</td>
<td>410</td>
<td>9</td>
<td>139</td>
<td>31</td>
</tr>
<tr>
<td>5</td>
<td>480</td>
<td>450</td>
<td>8</td>
<td>143</td>
<td>9</td>
</tr>
<tr>
<td>6</td>
<td>520</td>
<td>500</td>
<td>7</td>
<td>148</td>
<td>25</td>
</tr>
<tr>
<td>7</td>
<td>560</td>
<td>550</td>
<td>4</td>
<td>150</td>
<td>21</td>
</tr>
</tbody>
</table>
4.5. Cold deformation and annealing (without quenching and ageing)

The last method of CuFe2 alloy processing did not include quenching and ageing. The strips were cold rolled with rolling reduction 10%, 20%, 30%, 50% and 70%, then annealed at temperature of 480°C for 1, 2, 4, 6, 10, 12, 14, and 16 hours. Investigations into hardness (Fig. 19) and electrical conductivity (Fig. 20) were carried out after annealing.

![Hardness graph](image1)

**Fig. 19. Changes of hardness of CuFe2 alloy versus annealing time and rolling reduction; annealing temperature 480°C**

![Electrical conductivity graph](image2)

**Fig. 20. Changes of electrical conductivity of CuFe2 alloy versus annealing time and rolling reduction; annealing temperature 480°C**

The dependence of hardness on rolling reduction and ageing time at temperature 480°C run in a different way in this processing method. For 20% rolling reduction the hardness increased from 124 HV after 1 hour of annealing to 136 HV after 12 hours of annealing, then decreased to 132 HV after 16 hours of annealing. Hardness of the strip after 30% rolling reduction increased form 120 HV to maximal value of 140 HV after 10 hours of annealing. For higher values of annealing time hardness presented a decreasing tendency. A quite different phenomenon was observed during examination of CuFe2 alloy deformed with rolling reduction 50% and 70%. Hardness decreased monotonically from 135 HV (rolling reduction 70%) and 140 HV (rolling reduction 50%) to 120 HV (for both rolling reductions) after 16 hours of annealing. The reached hardness values were in the intended range 120-140 HV. However, the intended electrical conductivity was reached for samples with rolling reduction 70% after 8 hours of annealing at 480°C and for samples with rolling reduction 50% after 10 hours of annealing. Therefore, only this variants of processing method provided possibility to reach the intended functional properties. TEM investigations helped to explain this phenomenon.

![TEM image a)](image3)

**a)**

![TEM image b)](image4)

**b)**

![TEM image c)](image5)

**c)**

**Fig. 21. Microstructure of CuFe2 samples deformed with rolling reduction 20% and annealed at 480°C for 12 hours. TEM; a) dislocation clustering into cellular systems, b) dislocations inside iron γ precipitation, c) matrix coherent dispersive precipitations of iron γ**
Fig. 22. Microstructure of CuFe2 samples deformed with rolling reduction 30% and annealed at 480°C for 12 hours. TEM; a) matrix semi-coherent dispersive precipitations of iron γ, b) dislocations around precipitation of iron γ, c) twin of recrystallization growth.

Fig. 23. Microstructure of CuFe2 samples deformed with rolling reduction 50% and annealed at 480°C for 12 hours. TEM; a) dislocation free matrix area with globular iron precipitations, b) polygonization process, c) uniformly distributed iron precipitations in copper matrix, dark field.
Fig. 24. Microstructure of CuFe2 samples deformed with rolling reduction 70% and annealed at 480°C for 12 hours. TEM; a) polygonization process, b) polygonization process, c) recrystallization area, iron precipitations inside new grains and on twins boundaries, d) recrystallization area, iron precipitations inside new grains and on twins boundaries, e) area with different number of dislocations, f) blocking of dislocations on the fine dispersive precipitated iron particles.
Thin foil investigations revealed that in the cold deformed material with rolling reduction 20% and annealed at 480°C for 12 hours some ordering of dislocations structure into low energy order takes place (Fig. 21a). At this stage of the process, precipitation of globular iron γ particles (Fig. 21b) and presence of fine, coherent iron γ precipitations (Fig. 21c) was found.

The increase of rolling reduction up to 30% resulted in loss of coherence of globular precipitations (Fig. 22a). Fine iron γ precipitations still formed a barrier for the dislocations movement (Fig. 22b). Basing on TEM investigations, it can be concluded, that recrystallization process started in the defected matrix area, where recrystallization twins had formed and grown (Fig. 22d).

Cellular dislocations structure was observed in samples deformed with rolling reduction 50% (Fig. 23). Decrease of dislocations density and uniform distribution of iron precipitations in a copper matrix were seen.

For the rolling reduction of 70% (Fig. 24) cellular dislocation sets and recrystallization process were noticed in a structure. Nucleation of iron precipitations was observed in a matrix area and in twins boundaries. Dislocation free area were also seen.

5. Conclusions

Structure and properties of industrial CuFe² alloy differs significantly from the literature descriptions, especially after quenching process. Obtained results were influenced by industrial production conditions and the charging materials. It is worth to be mentioned, that alloy was prepared by melting in a channel-type induction furnace with acid lining and capacity 1500 kg. During melting no protective covering was used. Molten alloy was transferred by a runner to the foundry furnace. This furnace was equipped with continuous casting mould for horizontal casting of 15 mm x 400 mm strip. The equipment consisted of graphite mould mounted in a copper cooler with steel housing. This method of casting caused faster cooling of smaller volume of liquid metal in a time unit, as opposed to the semi-continuous casting of ingots of greater cross-sections. It could be assumed, that the dissolved in a time unit, as opposed to the semi-continuous casting of ingots of greater cross-sections. It could be assumed, that the dissolved in a a matrix area, where recrystallization twins had formed and grown (Fig. 22d).

Cold deformation after casting (before quenching) significantly influenced the inhomogeneous microstructure changes after quenching (there is no hot rolling after casting in this technology). Cold deformation with rolling reduction 30% and 50%, before quenching and ageing at temperature 500°C for 420 minutes, provided possibilities to reach (maximal in this processing method) electrical conductivity 34 MS/m and hardness 100 HV.

Cold deformation after quenching and ageing provided possibilities to reach (maximal in this processing method) electrical conductivity 34 MS/m and hardness 113 HV.

Quenching and ageing of industrial CuFe² alloy results in (maximal in this processing method) electrical conductivity of 36.5 MS/m and hardness 106 HV.

Cold deformation with rolling reduction 70% and annealing at temperature 480°C for 12 hours provided possibilities to reach maximal electrical conductivity 37 MS/m and maximal hardness 136 HV.

The presented investigation results, besides their cognitive values, provide many useful information which might be implemented in a industrial practice.

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References