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Effect of two-stage isothermal annealing on microstructure CuAl10Fe5Ni5 bronze with additions of Si, Cr, Mo, W and C

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Abstract

The aim of this study was to investigate the effect of a two-step isothermal annealing respectively at 1000 ˚C for 30 min, then at the range of 900÷450 ˚C increments 50 ˚C on the microstructure CuAl10 Ni5Fe5 bronze with additions of Si, Cr, Mo, W and C, cast into sand moulds. The study concerned the newly developed species, bronze, aluminium-iron-nickel with additions of Si, Cr, Mo, W and C. In order to determine the time and temperature for the characteristic of phase transitions that occur during heat treatment of the test method was used thermal and derivation analysis (TDA). The study was conducted on cylindrical test castings cast in the mould of moulding sand. It was affirmed that one the method TDA can appoint characteristic for phase transformations points about co-ordinates: τ (s), t (˚ C), and to plot out curves TTT for the studied bronze with their use. It was also found that there is a five isothermal annealing temperature ranges significantly altering the microstructure of examined bronze.

Keywords: Innovative materials and casting technologies, Technological properties, Aluminium-nickel-iron bronze, Microstructure, TTT curves

1. Introduction

It results from the analysis of the current condition of knowledge, that the characteristics of the isothermal decomposition of β phase were worked out for aluminium bronzes on the basis of conducted investigations for bronze CuAl11, 3 and CuAl9Fe3 [1,2]. The characteristics TTT of these bronzes are shown in Figures 1 and 2. It results from them that make addition to the aluminium bronze of the iron influences (Fig. 1 and 2):

- enlargement of the stability of phase β by quick cooling down,
- decrease of the range of temperature and the time of the partial transformation of β phase in α phase ,
- extension of the range of the temperature and decrease of the range of the time of duration of the transformation $\beta \rightarrow \beta_1$,
- enlargement of the area of the durability of β_1 phase ,
- extension of the range of temperature and the time of the decomposition of β_1 phase on eutectoid ($\alpha+\gamma_2$),
- the lowering of the temperature of Ms from 450 °C to 385 °C.

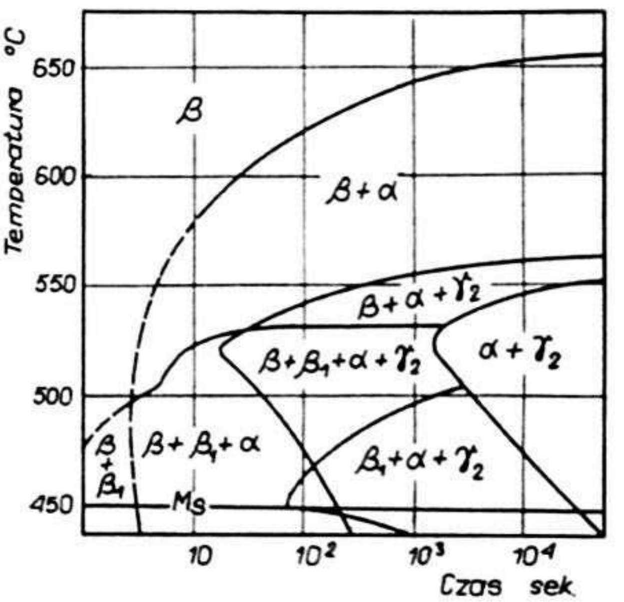


Fig. 1. Graph isothermal decomposition β phase for Cu-Al alloy containing 11.3% Al [1]

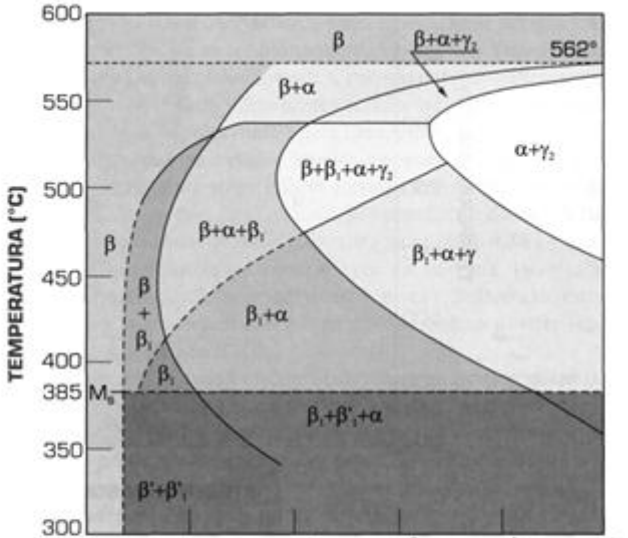


Fig. 2. Graph TTT isothermal decomposition β phase during the cooling copper alloy with aluminium CuAl9Fe3 (according Prowans S.) [2]

Make additions of 3% iron to the aluminium bronze therefore not influenced to change the type formed in the isothermal annealing phases, only resulted primarily the TTT characteristics move towards longer times and lower temperature phase transitions. To date, no published study on the phase transformations occurring during isothermal annealing aluminium-nickel-iron bronze with additions of Si, Cr, Mo, W and C. Therefore, the aim of the study was to examine effect a two-stage isothermal annealing, respectively, at 1000 ˚C, and then in the temperature in the range 900÷450 ˚C increments 50 ˚C, on the microstructure CuAl10Ni5Fe5 bronze with additions of Si, Cr, Mo, W and C, cast into sand moulds. The studies resulted in the development of new grades of aluminium-iron-nickel bronze additives alone or in combination of Si, Cr, Mo, W and/or C [3÷10].

The acquaintance of the TTT characteristics will make possible of the proper selection of the parameters of the thermal processing in the aim of steering microstructure, and in the consequence proprieties after the isothermal processing thermal of new elaborated bronzes.

2. Research methodology

The study was conducted on CuAl10Ni5Fe5 bronze with addition 0.19% Si, 0.39% Cr, 0.03% Mo, 0.01% W and 0.48% C. The shape and dimensions of the test sample microstructure and TTT curves are shown in Figure 3

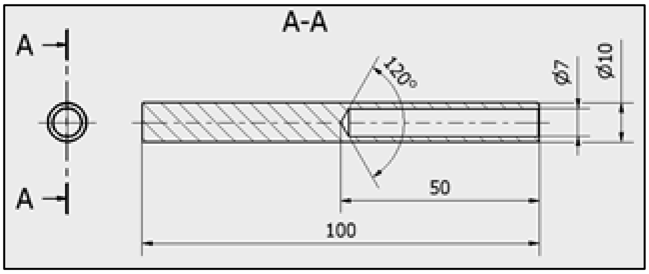


Fig. 3. The shape and dimensions of the test sample microstructure and TTT curves

Heat treatment process schematically shown in Figure 4.

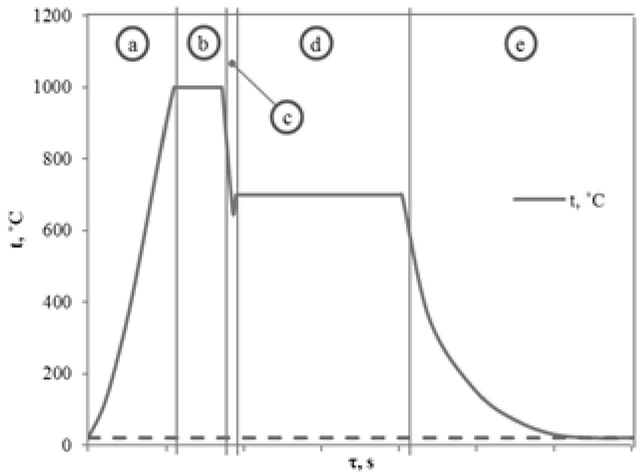


Fig. 4. Isothermal two-stage process of heat treatment:
Stage I - a) warming, b) isothermal annealing of 1000 ˚C;
Stage II - c) cooling the ambient air or water,
d) the isothermal annealing of (900÷450 ˚C),
e) cooling in ambient air

The thermal processing of the bronze was consisted from processes:

Stage I:

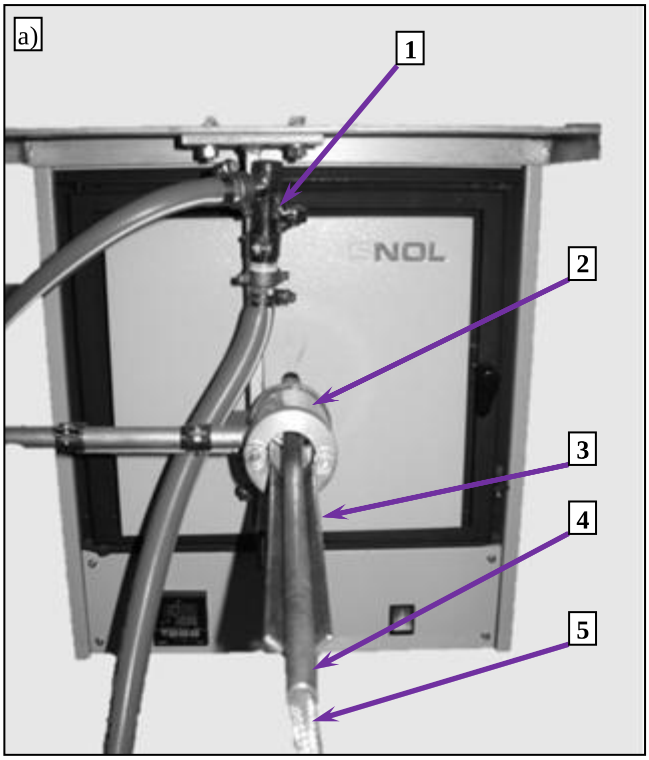
- warming to the temperature 1000 ˚C,
- annealing (saturating) in the temperature 1000 ° C and time 30 minutes,

Stage II:

- cooling in ambient air or (intensive cooling) water to the temperature of isothermal warming,
- isothermal annealing in the temperature 900 ℃ ÷ 450 ℃ with grading 50 ℃ till the moment of the end of phase transformation - not longer than 7 days,
- cooling down in the air to the temperature of surroundings.

The heat treatment was performed on a test stand shown in Figure 5. The stand is composed of two types of furnaces Snol 1100. Separately in each of them adjusted to the desired temperature, according to the stage of heat treatment.

Change in temperature of the tested bronze samples attached to a lance (Fig. 5a), during heat treatment, measured by thermocouple type S (Pt-PtRh10) inserted into the hole Ø7x50 (Fig. 3) and recorded on a computer using the devices Crystaldigraph.



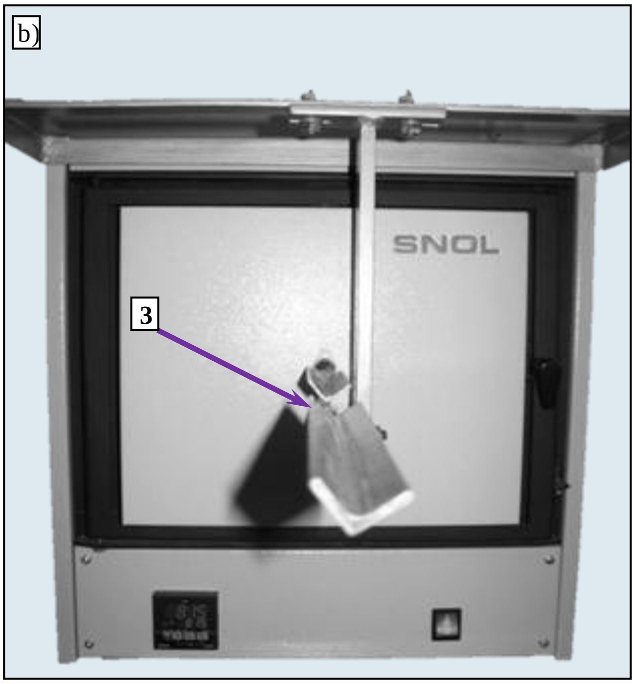


Fig. 5 The test stand:
a) furnace for Stage I of the isothermal annealing (1000 ˚C):
1 - water cooling system valve, 2 - cooling chamber with water,
3 - lead channel, 4 - lance (sample holder),
5 - thermocouple compensation cable type S,
b) furnace for Stage II isothermal annealing (900÷450 ˚C)

With the use of the TDA program, it was possible to determine characteristics:

- temperature change curve t=f(τ), ˚C;
- derivation of the curve (kinetics of thermal processes) dt/dτ=f '(τ) ˚C/s.

ATD identified in the curves characteristic points of thermal effects occurring during the two-stage isothermal annealing, respectively:

- point I - start of κ phase crystallization,
- point II - start of α phase crystallization,
- point III - start of eutectoid ($\alpha+\gamma_2$) transformation,
- point IV - end of eutectoid ($\alpha+\gamma_2$) transformation,
- point V - start of phase β_1 crystallization,
- point VI - start of peritectoid ν phase crystallization,
- point VII - end of peritectoid ν phase crystallization,
- point VIII - end of β_1 phase crystallization.

Metallographic specimen were prepared on the samples forehead (Fig. 3). The sample etched reagent Klemm II. Digital images of the microstructure was performed on the Nikon Eclipse microscope.

3. The results

In Figure 6 (a÷e) shows representative temperature changes curves $t=f(\tau)$, bronze CuAl10Fe5Ni5SiCrMoWC, recorded during two-stage isothermal annealing at 1000 ° C for 30 min, followed by annealing at a temperature of 750 ° C.

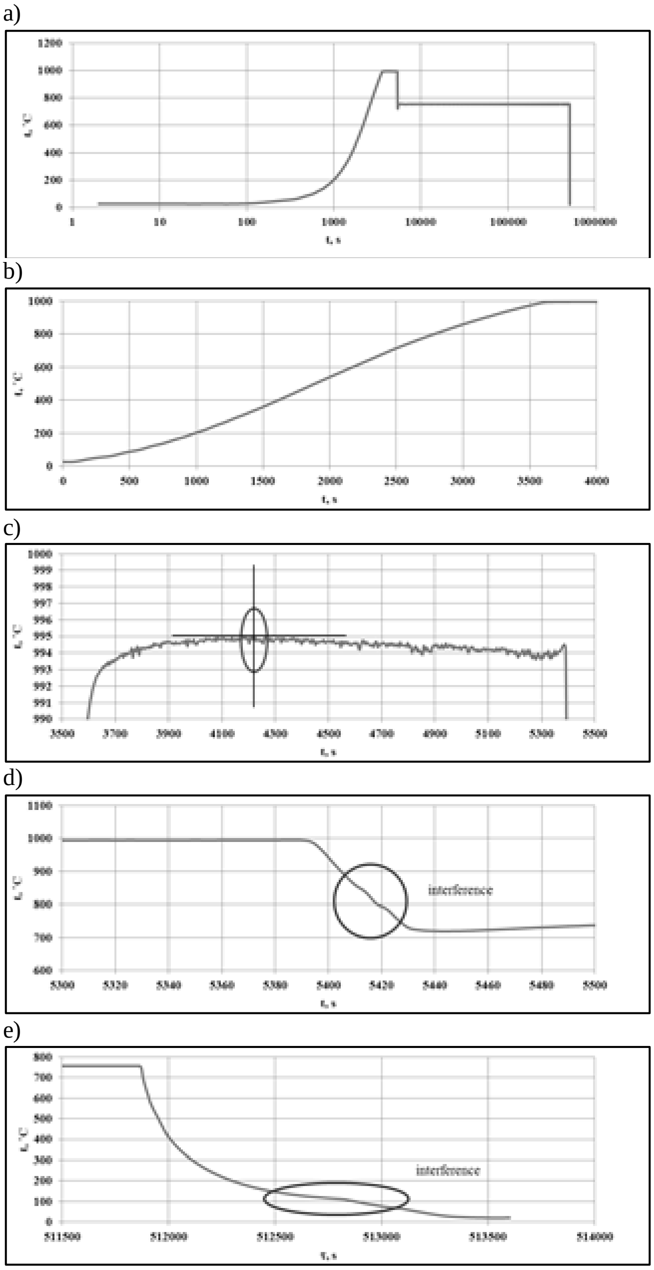


Fig. 6. The curves $t=f(\tau)$ changes in temperature of bronze CuAl10Fe5Ni5SiCrMoWC during two-stage annealing (Stage I - 1000 ˚C, Stage II - 750 ˚C):
a) the curve $t=f(\tau)$ for the whole heat treatment process, b) warming to a temperature of 1000 ˚C, c) annealing of at 1000 ˚C for τ = 30 min, d) cooling to a temperature of 750 ˚C
e) cooling in ambient air

In Figure 7 (a÷e) shows the curve $t=f(\tau)$ changes in temperature and the curve of derivational $dt/d\tau=f'(\tau)$ with the designated characteristic points, for taking the CuAl10Fe5Ni5SiCrMoWC bronze phase transitions, registered during two-stage isothermal annealing.

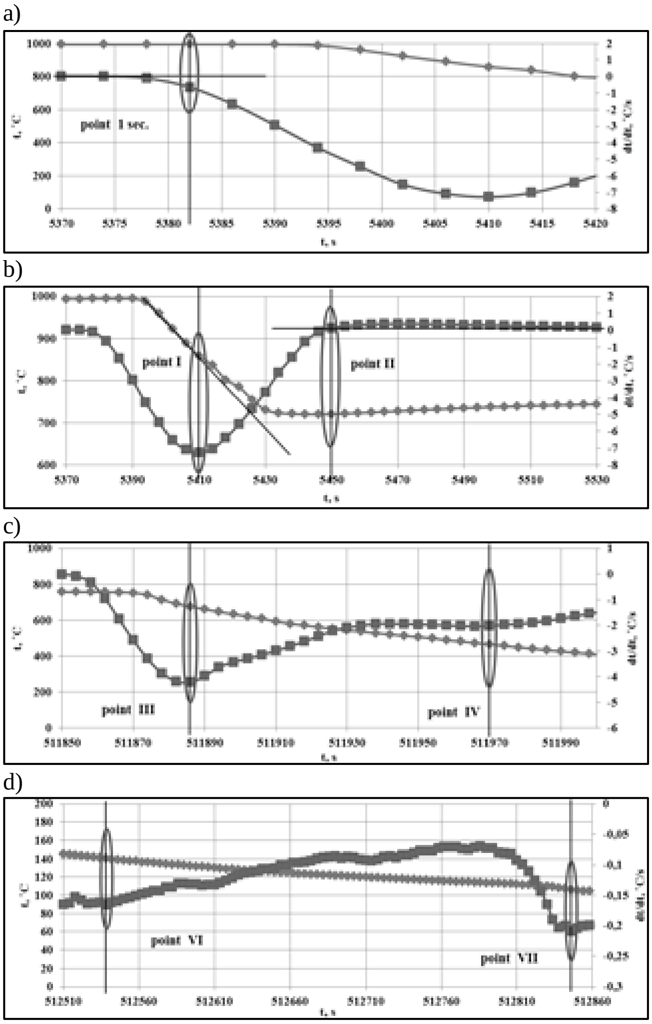


Fig. 7 The curves $t=f(\tau)$ ◆) changes in temperature, and $dt/d\tau=f'(\tau)$ ■) the derivative for the CuAl10Fe5Ni5SiCrMoWC bronze during two-stage annealing
(Stage I - 1000 ˚C, Stage II - 750 ˚C):
a) identification of so-called point. "1-st second cooling"
b) identification of points I and II,
c) identification of points III and IV,
d) identification of points VI and VII

Microstructure of CuAl10Fe5Ni5SiCrMoWC bronze in the cast state and after a two-stage isothermal annealing is shown in Figure 8.

On the curve of temperature changes in the Stage I of the isothermal annealing (1000 °C) temperature extremum have been identified local (Stage I, Fig 6 c). Identified maximum temperature (in spite of the isothermal heating process) probably indicates that since the maximum registration begins in the bronze

process of dissolution of κ phases in β solid solution, hence the decrease of the temperature (heat absorption necessary to dissolve in the β solution κ phase). On the curve of temperature changes $t=f(\tau)$ identified several thermal effects of phase transitions. These effects (interference in the course of temperature changes) were identified during cooling bronze to the temperature of isothermal annealing and the initial period of annealing at 750 ˚C (Stage II, Fig. 6 d) and during cooling in surrounding air (Stage II, Fig. 6 e).
After completing the first stage of annealing, using the first derivative of temperature over time $dt/d\tau=f'(\tau)$, defined the starting point cooling bronze to the second stage of annealing temperature. Fixed point on the derivational curve (time from start of measurement) was assumed as a so-called "first seconds of cooling" (Fig. 7 a, 1 sec. for the current time $\tau b=5382$ s). Appointment of this point, and above all the corresponding τb current time, for all samples, respectively, for various temperature levels in Stage II of isothermal annealing, enabling the graph of TTT characteristics for the test of the bronze after heat treatment cycle. These characteristics are plotted in a logarithmic time axis system, the smallest unit of time equal to 1 s.
At the stage of cooling bronze around 1000 °C to about 750 °C, were identified on the curve derivational point setting the beginning of the re-crystallization of the phases κ - a point I (Fig. 7b, $t_I=872$ °C, $\tau b=5410$ s, $\tau_I = 28$ s) and partial transformation of β phase in α - point II (Fig. 7b, $t_{II}=720$ °C, $\tau b=5450$ s, $\tau_{II}=68$ s).
During bronze cooling to ambient temperature, ie after removing the sample from the furnace and cooling in surroundings air on the curve derivational thermal effects have been identified since the beginning of the transformation phase eutectoid $\beta \rightarrow \alpha+\gamma_2$ - a point III (Fig. 7c, $t_{III}=675$ °C, $\tau b=511886$ s, $\tau_{III}=506504$ s) and thermal effects of the end of phase $\beta \rightarrow \alpha+\gamma_2$ eutectoid transformation - point IV (Fig. 7c, $t_{IV}=473$ °C, $\tau b=5118966$ s, $\tau_{IV}=506584$ s). Thermal effects is also registered $(\alpha+\gamma_2)$eutectoid onset in ν phase peritectoid - point VI (Fig. 7d, $t_{VI}=141$ °C, $\tau b=512538$ s, $\tau_{VI}=507156$ s) and its the end of - point VII (Fig. 7d, $t_{VII}=101$ °C, $\tau b=512874$ s, $\tau_{VII}=507492$ s).
Identified by this method the temperature and time of the characteristic thermal effects, for the corresponding temperature of isothermal annealing Stage II study bronze shown in Table 1.

In the cast state microstructure of the CuAl10Fe5Ni5SiCr-MoWC bronze was folded from following phase (fig. 8):

- the primary dendritic of intermetallic phase $\kappa_{Fe(NiSiCrMoWC)}$,
- small intermetallic phaze $\kappa_{Fe(Ni)}$,
- comparatively thick phaze α
- and eutectoid $\alpha+\gamma_2$.

A two-step annealing bronze at t=1000 °C and 750 °C resulted in the following changes in its microstructure (Fig. 8):

- experienced a partial coagulation of primary dendritic $\kappa_{Fe(NiSiCrMoWC)}$ phase
- $\kappa_{Fe(Ni)}$ phase crystallized, relatively small and evenly distributed in the microstructure of bronze in the shape of spherical precipitates,
- crystallized α phase, and $(\alpha+\gamma_2)$ eutectoid in the form of small platelet precipitates,
- part of eutectoid $(\alpha+\gamma_2)$ turned into peritectoid phase ν.

Microstructure of the test obtained in the bronze two-step of isothermal annealing first at 1000 ˚C for 30 min. (Stage I), then at 900÷450 ˚C, with increments 50 ˚C (Stage II) has several changes (Fig. 8). In assessing the resulting microstructure of bronze, it was observed that for Stage II annealing temperature equal to t = 900 ° C was obtained microstructure very similar to the microstructure of bronze in the cast state. What distinguishes the microstructure of heat treated bronze from bronze in the cast state is a form and size of intermetallic phases $\kappa_{Fe(SiNiCrMoWC)}$. After annealing, the phases of κ have partial spheroidization, increased their size at the cost of quantity.
Characteristic of the heating process at Stage II, 850 and 800 ºC is to create a bronze in the microstructure, in addition to platelet precipitates, large particles α phase of irregular shape.
In the process of annealing in Stage II, in the temperature range 750÷600 ºC, the microstructure of the test bronze creates a fine plate $(\alpha+\gamma_2)$eutectoid. The degree of fragmentation of platelets $(\alpha+\gamma_2)$ eutectoid increases with decreasing annealing temperature.
In the case of heating the CuAl10Fe5Ni5SiCrMoWC bronze in temperature 800÷750 ºC peritectoid reaction takes place resulting in the partial decomposition occurs (α+γ2) eutectoid on ν phase peritectoid.
As a result of annealing in Stage II in the temperature range 600÷450 ºC in the bronze microstructure obtained eutectoid $(\alpha+\gamma_2)$ with a characteristic globular structure. This is probably due to the presence in the microstructure bronze before transformation eutectoid ordered β_1 phase.

Figure 9 shows of isothermal TTT diagram decomposition β phase during cooling CuAl10Fe5Ni5SiCrMoWC bronze. Make addition to the aluminum bronze Fe, Ni, Si, Cr, Mo, W and C received, compared to existing charts TTT (bronzes: CuAl11, 3, CuAl9Fe3) to:

- creation lines start crystallization of phases $\kappa_{Fe(Ni)}$ (about 900 ˚C),
- increase the line (temperature), start the partial transformation of β phase in α and the shift towards longer times,
- a significant shift towards longer decomposition times of start the phase β (β1) at the eutectoid $(\alpha+\gamma_2)$,
- increasing the temperature disordered β phase transition in an β_1 ordered,
- creation the starting line transformation eutectoid $(\alpha+\gamma_2)$ in peritectoid phase ν.

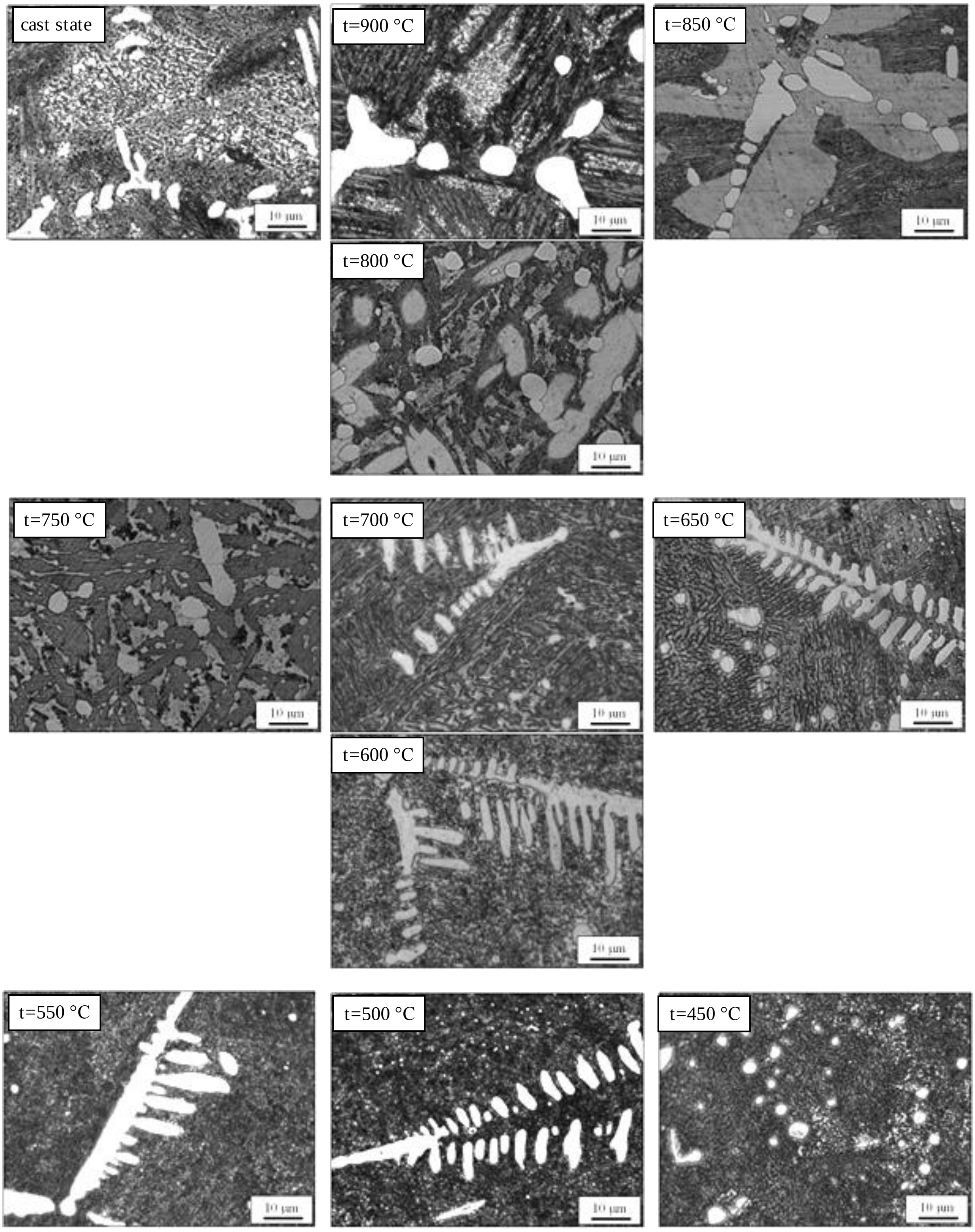


Fig. 8. Microstructure of bronze CuAl10Fe5Ni5SiCrMoWC in the cast state and after heat treatment

Table 1.
Temperature t (˚C) and time τ (s) from 1-st sec. cooling characteristic phase transitions during isothermal annealing CuAl10Fe5Ni5SiCrMoWC bronze in the temperature range 900÷450 ˚C

II Stage	$\beta \rightarrow \beta+\kappa_{Fe(Ni)}$		$\beta \rightarrow \beta_1$ (s)		$\beta(\beta_1) \rightarrow \alpha$		$\alpha+\gamma_2$ (s)		$\alpha+\gamma_2$ (f)		$\alpha+\gamma_2 \rightarrow v$ (s)		$\alpha+\gamma_2 \rightarrow v$ (f)		$\beta \rightarrow \beta_1$ (f)	
t_annealing, °C	time from the first second of cooling, τ, s	t, °C	time from the first second of cooling, τ, s	t, °C	time from the first second of cooling, τ, s	t, °C	time from the first second of cooling, τ, s	t, °C	time from the first second of cooling, τ, s	t, °C	time from the first second of cooling, τ, s	t, °C	time from the first second of cooling, τ, s	t, °C	time from the first second of cooling, τ, s	t, °C
900	20	929			492300	814	492400	464								
850	25	908			429341	779	429381	623								
800	44	872			108	481	584528	656			584828	313	585344	124		
750	28	859			68	720	506504	675	506584	473	507156	141	507492	101		
700	17	901			37	640										
650			4	407	32	712	402572	644								
600			20	517	64	322	357944	603								
550			36	520			327193	553								
500			20	582	192	314	108858	496							186258	495
450			28	477	48	292	418110	450							143	268

(s) – start of transition
(f) – finish of transition

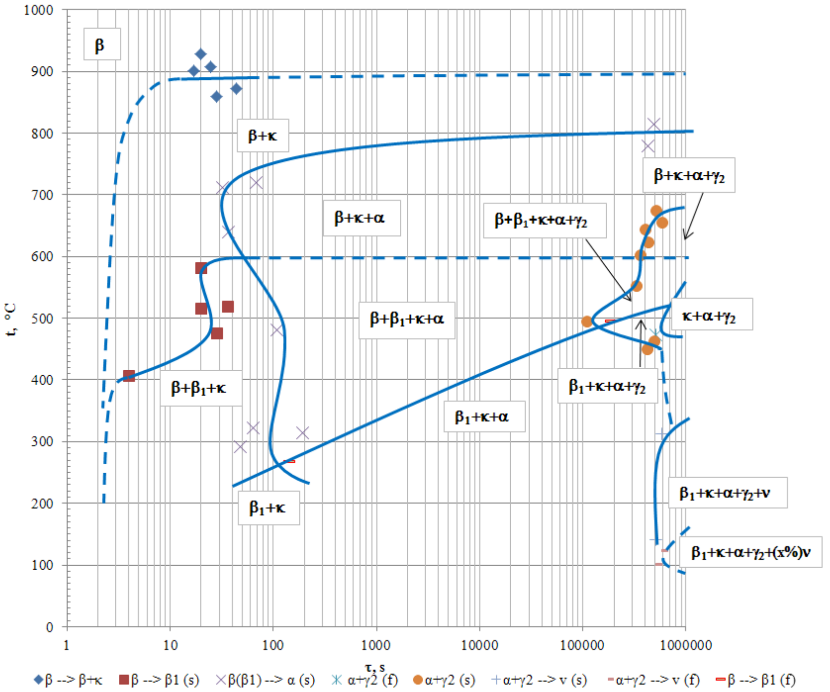


Fig. 9. Graph TTT isothermal decomposition of β phase during cooling CuAl10Fe5Ni5SiCrMoWC bronze, (s) start transition, (f) finish transition

4. Conclusions

The study indicates the following conclusions:

- method of thermal and derivation analysis (TDA) can identify thermal effects (characteristic points - time and temperature) associated with phase transformations during heating and cooling bronze,
- characteristic points obtained allow to determine characteristics of TTT for the test bronze,
- two-step annealing bronze causes coagulation and coalescence of intermetallic κ phases,
- with the change of the second stage annealing temperature is achieved, compared to the microstructure of bronze in the cast state (beyond of coagulation and coalescence of intermetallic phases κ), following changes in the microstructure:
- 900 °C – no change,
- 850 – 800 °C – α phase separation in the form of large irregular grains,
- 800 – 750 °C – creation in the microstructure, by the peritectoid reaction ($\alpha+\gamma_2 \rightarrow \nu$), phase ν,
- 750 – 650 °C – create platelet eutectoid ($\alpha+\gamma_2$),
- 600 – 450 °C – create globular eutectoid ($\alpha+\gamma_2$).

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