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# Investigation of the Structural Characteristics of Amorphous and Nanocrystalline Copper - Loaded Solders

# II. Experimental Part

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**Abstract**

Structural changes of samples of crystalline, amorphous and nanocrystalline copper-loaded solder with additions of nickel, tin and phosphorus produced under factory and experimental technologies has been studied. Analysis of structural changes of melt taking place during melting and amorphization has been performed in paper I [1]. A differential thermal analysis of this samples has been conducted. A two-step transition of amorphous structure into nanocrystalline one was discovered. During heating thermal effect of samples produced under experimental smelting technology turned out to be 20 per cent higher, which is evidence of a more complete amorphization of alloys.

**Keywords**: copper-loaded solder, amorphous ribbons, differential thermal analysis, thermal effect

## Introduction

The mechanism of the micro-structure changes of liquid metal at melting has been discussed in the first part of paper [1]. Structural-phase state of the material is determined not only by the interaction of chemical elements, but also by alloy production conditions [1]. In the process of melting the formed liquid metal is characterized by non-equilibrium and inhomogeneous state. After melting the unstable state of the melt persists for a long time in a wide temperature range, up to a certain temperature $t_c$.

When heated to such a temperature the melt structure is changing rapidly. Reliability of structural change is confirmed anomalous behavior of the physical properties of the melt [2-4].

## Main Part

The differential thermal analysis was used to study the processes of transformation during melting, solidification and other structural changes of copper solder in the liquid and solid states. Appearance of deviations on the DTA curves such as peaks and inflections reveals structural transformations, and thermal effect describes their intensity.

The test samples were melted out of pure components in an electric furnace. Amorphous ribbon solder was manufactured by single-roll spinning method. DTA experimental results of four samples, which are similar in composition, vary slightly, and therefore in the figures 1-3 only typical dependencies are shown.

In the process of studying the impact of maximum heating temperature ($t_{heat}$) on DTA curves there was used the information, which was published in work [5] about the value of critical temperature ($t_c$), heating up to which contributes to the formation of the equilibrium and the homogenous melt state.

Figure 1a shows that when melting the copper-loaded solders a drastic change in the structure of the crystalline sample occurs, and then at other four temperatures, there are changes in the status of the melt. The presence of small deviations on DTA curve is confirmed by statistical data set and shows different thermal stability of structural elements of the liquid solder.

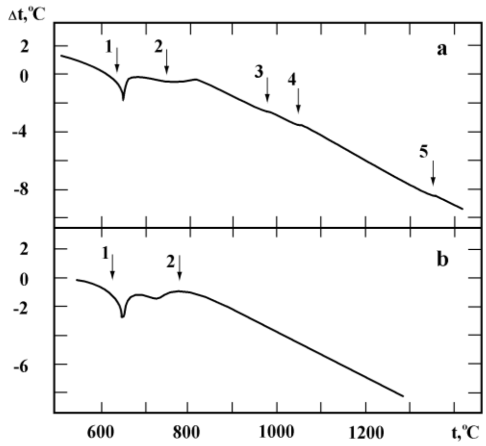


**Figure1.** DTA curves of samples of copper-loaded solder during heating:
a-original condition; b- repeated research of sample «a»;
тumerals - deviations from the classical dependence

After heating to 1320°C, i.e. above $t_c$ crystallized samples were repeatedly used for the DTA and it was found out that the thermal effect (Δt) at $t_L$ was significantly higher, a maximum of 2 shifted towards higher temperatures and deviations 3, 4 and 5 were missing. (fig. 1b)

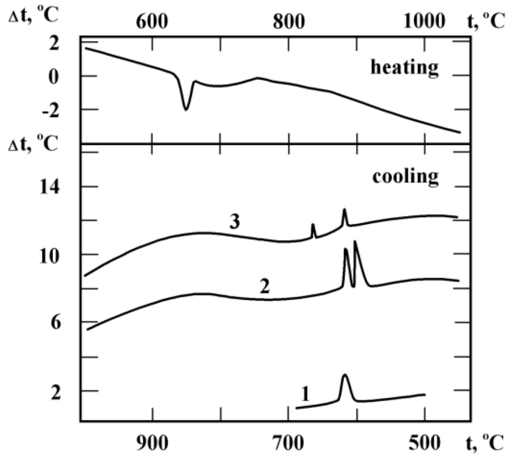


**Figure 2.** DTA curves of samples of copper-loaded solder heated to temperatures:
1-700°C, 2 – $t_c$+250°C; 3- - $t_c$+400 °C

Figure 2 shows a complex influence of $t_{heat}$ on DTA curves and Δt value. With increase of $t_{heat}$ thermal effect increases in the entire temperature range of the study and considerable changes are taking place in a molten state near the solidification temperature and phase transition.

The results of studies of amorphous (a) and nanocrystalline (b) films manufactured from the melt, which was heated during the melting process below $t_{k,}$ are marked by the index 1, and above the $t_c$ - by the index 2 (fig. 3). The peaks on the DTA curve reflect a two-step transition of amorphous structure into nanocrystalline one.

The first stage appears to be linked to the formation of nanocrystals in clusters. Whereas the amorphous intercluster space is the limiting factor to increase of the crystal size. At the second stage the structurally amorphous intercluster volumes become nanoclusters. The thermal effect of these processes occurred to be 20% higher in samples of test technology of melting, which is evidence of a more complete amorphization of alloys. The same result was obtained during the study of nanocrystalline samples 1 b and 2 b (fig. 3).

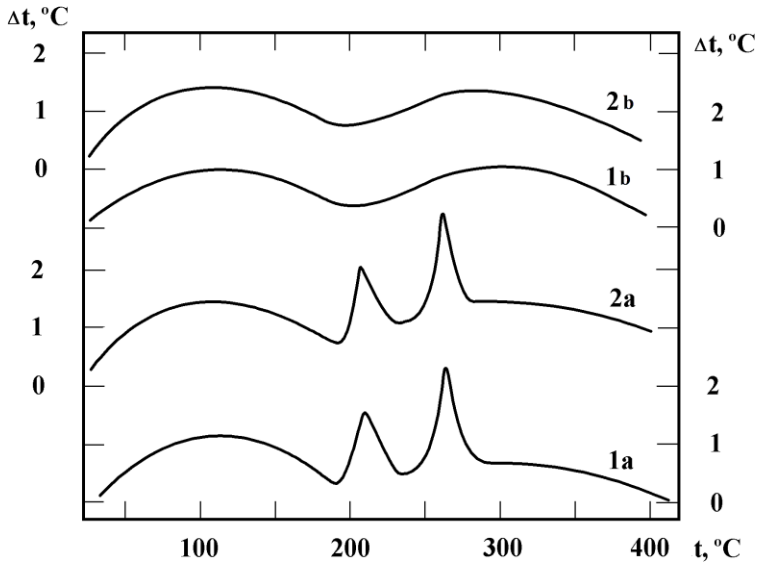


**Figure 3.** Influence of melt preparation before spinning process on DTA curves view and thermal effect (Δt) of structural changes of samples of copper-loaded solder: 1 and 2- smelting on traditional and experimental technology; a- amorphous sample, b- nanocrystalline sample.

## Conclusion

A differential thermal analysis of samples of crystalline and amorphous and nanocrystalline copper-loaded solder with additives of nickel, tin and phosphorus produced under factory and experimental technologies has been conducted. The equilibrium and homogeneous state of the liquid copper-loaded solders before spinning process contributes to forming a structure of solid amorphous and then nanocrystalline material with high energy and dissipative characteristics. The last can increase output of technologically suitable amorphous ribbon determined by mechanical flexibility proof testing.

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