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The effect of outside energetic treatment on the process of crystallization analyzed by AE-method and on the stabilization of melting plateau

S.Prokhorenko[a], V.Prokhorenko[a], S.Mudry[b], W.Halczak[b], A.Panas[c]

[a]Information Measuring Technique Department, National University „Lvivska Politechnika“, 12 Bandera St., Lviv, 290646, Ukraine.

[b]Physics of Metals Department, Ivan Franko Lviv National University, 8 Kyrylo and Mephodyi St., Lviv, 290005, Ukraine.

[c]Aviation Technology, Military University of Technology (WAT), Kaliskiego 2, Warsaw 00-908 Poland

Results of studying of melting and crystallization processes of metal melts previously handled by exterior pulsing fields are presented. Vibration treatment causes the formation of metal with grain structure of small size that corresponds to more homogeneous melt before the crystallization front. Satisfactory conformity between recorded features of AE signals and level of homogeneity of melt before crystallization takes place. The viscosity measurements confirm the inhomogeneities in the melt before solidification. The effect of the vibration treatment on the improvement of melting plateau stability is studied.

1. INTRODUCTION

At the construction of temperature reference points the eutectic alloys have been considered effective lately. In order to have the crystallization temperature stabilized (as well as the melting one) the homogeneous state of the melt and isotropic temperature field are needed. In particular, the tendency of eutectic to gravitational sedimentation and micro-separation is evidently proved and connected with this problem [1].

The structure of eutectic melts is more complicated than that of pure metallic liquids. At the temperatures near liquids curve the structure of melts is determined by existence of fractal formations (FRs) [2]. FRs are different from crystalline nucleus in the change of dimensions from 3 in the center to 2 at interface boundary. Taking into account the short period of their existence time, the fractal formations can be considered dynamical groups of alternative/inhomogeneous topological dimension.

On the basis of modern physical vision it is possible to state that solid state structure formed upon crystallization is the structure of melt anticipating the phase transition. FRs in this case is the determinant of number, size and complexity of dendrites. The branches of FR determine the inhomogeneity of diffusion before crystallization front and, as a result, the instability of crystallization temperature.

In the case of applying of outside non-monotone energetic influence it is possible to make the conditions which promote the „comminution" of melt structure that should improve the temperature stability.

In order to check the effectiveness of such influence on the melt we used the fixation of acoustical emission method (AE) in the crystallization process. The possibility of checking the structure without the deconstruction of temperature reference point cell is the advantage of this method. Radiation was registered within the interval 0.1÷1.5 MHz. The neareutectic Sn97Cu3 alloy was chosen as a model.

As experimental confirmation of the influence of fractal formations on the structure we measured the cinematic viscosity coefficient (CVC). The viscosity coefficient was measured using rotating cylinder method. A cylinder crucible, which is filled by investigated liquid, was placed in a vacuum chamber. Damped oscillations were analyzed in order to calculate the CVC.

2. RESULTS AND DISCUSSION

Acoustic Emission and Vibration Treatment

The goals of experimental studies were:

-1- To determine AE-radiation parameters which should be in correlation with grain size in the structure of solidificated melt and as a result, with its homogeneity.

-2- The determine the duration of the influence of external energetic field on the melt.

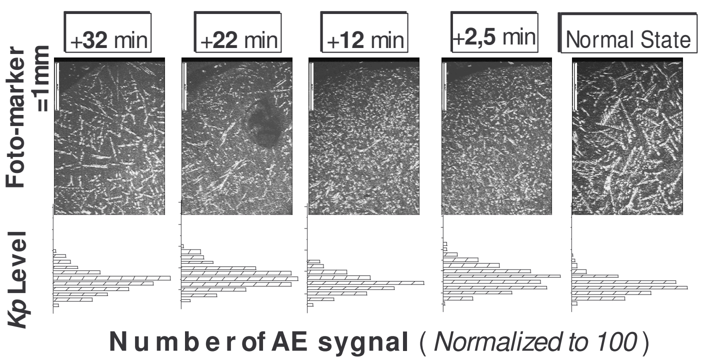

Figure 1. The correlation between histogram *Kp* and the structure of solidificated melt as a function of time since the termination of vibration treatment

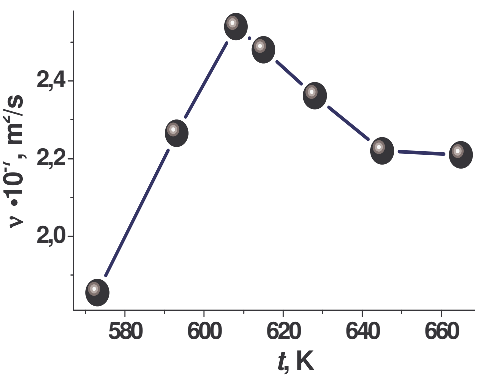

Figure 2. The viscosity of tested $Sn_{0.97}Cu_{0.03}$ alloy

The external influence was realized by vibration treatment of 49Hz frequency and 1.5 min duration.

Parameter *Kp* was chosen as a correlation parameter of acoustical emission. This parameter describes the acceleration in structure change process of researched material.

Fig.1 shows the correlation between *Kp* histogram (he number of signals of the same *Kp*) and structure of crystallized alloy depending on the time passed since the termination of vibration treatment. AE- signals were recorded during the crystallization process period.

The fractal description of structure.

The near eutectic alloy of this concentration was studied by means of viscosity measurement method. This physical characteristic is of great interest due to its importance for structure changes with temperature. The segregation of atoms before solidification leads to the increasing in CVC. It is possible to assume that viscosity coefficient for tested alloys should be somewhat higher than one for liquid tin. This assumption can be true in the case of microhomogeneous solution, close to ideal one. But taking into account the structural data on the existence of inhomogeneous atomic distribution in binary eutectic melts, this assumption is not quite motivated.

As it follows from our data the viscosity coefficient is lower than one for pure liquid at low temperatures. With further heating it shows anomalous behavior. Instead of decreasing its temperature dependence it increases to some point and then typical decreasing follows. Thus the λ-like dependence is observed indicating the deviation from exponent one. This deviation is caused by the deviation departure in concentration of alloy from eutectic one. Thus, within some temperature range the mixture of liquid and crystalline phase exists that is the reason for such anomalous behavior.

Viscosity was measured in cooling regime. It is important at the temperatures near eutectic one because the metastable phases formed at rapid cooling can change the values of CVC.

Thus the viscosity measurement method can be used for testing liquid before solidification. If we have purely eutectic melt the temperature dependence of viscosity coefficient shows exponential dependence. In other cases this dependence will be transformed into λ-like one.

In some cases liquid eutectic alloys can display local deviation of concentration. Such liquation of components can cause the change of solidification mechanism. This and many other structural changes can be effectively estimated using viscosity data. The rearrangements of atoms in fractal structure of binary liquid eutectic before its transition to solid state makes its contribution to temperature dependence of viscosity coefficient.

The effects of outside energetic treatment on the stability of melting plateau.

In-Ga-Sn melt of eutectic concentration has been studied within the project on building low-temperature reference point based on eutectic (used melt volume: $13cm^3$). Composition (in mass%): 66.960Ga; 20.497In; 12.543Sn. Purity of initial metals was at least 5N. Stabilization temperature: 10,587±0,003°C. We studied the expediency of applying outside energetic treatment of the alloy used before its crystallization for the stabilization of temperature/time characteristics of melting plateau (temperature of beginning/termination of the process, stability of temperature reproduction within the limits of the plateau).

Platinum thermometer OP2530 (Pt100, class 1/3B) placed in the well of reference point cell was used for measuring temperature (see *fig.3*). Temperature recorder is "Keithley 2700". Sampling interval: 1.5s.

Energetic treatment {VT5} was carried out by 5-minute ultrasonic oscillation with frequency 2.65 MHz in the melt with the temperature 15°C higher than the melting point. Change of the temperature dependence curve shape is shown in *fig.4.* Improvement of melting plateau stability is seen on the enlarged section. Reduction of overcooling before alloy crystallization after VT5 is considerable. It enabled us to use less powerful (more mobile) cooling system increasing the mobility of the whole reference point.

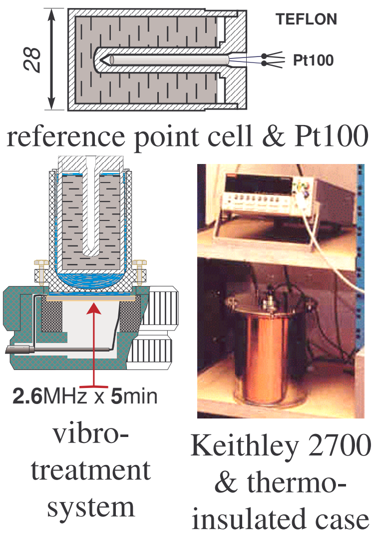

Figure 3. Components of experimental complex

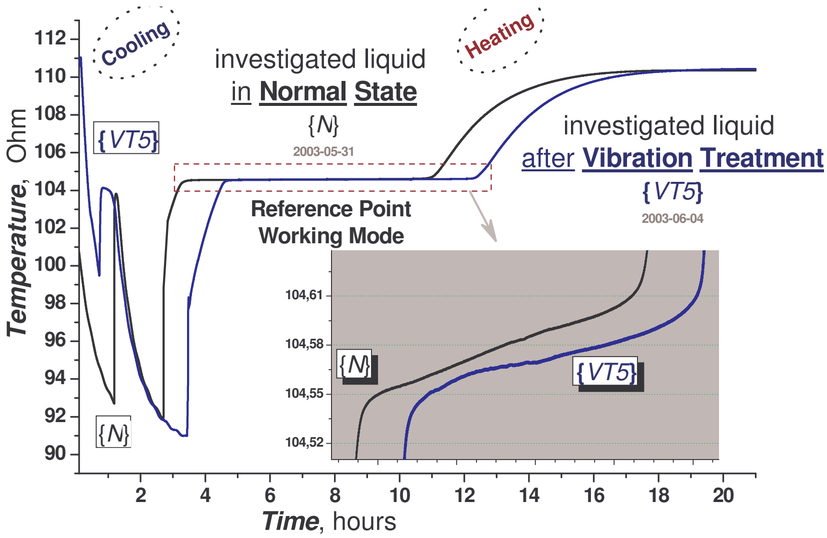

Figure 4. Obtained results (with enlarged view of the melting plateau). {*N*}- normal working mode, {*VT5*} - working mode with In-Ga-Sn eutectic melt after vibrotreatment.

3. CONCLUSION

The paper presents suggestions and experimental confirmation of positive effect of vibration on the stability of temperature reference point performance. Non-linearity of Sn97Cu3 melt structure temperature transformation is demonstrated with the use of viscosity measurement method. Method of acoustic wmission for controlling the structure formation after melt vibrotreatment has been approved.

REFERENCES

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