Differential thermal analysis of the results of isothermal discrete scanning of sheet aluminum material

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Abstract. The article presents an application of temperature and thermal analysis of discrete scanning data of heat perception of alloy 1163 to take into account thermal effects during plastic deformation.

1. Introduction

Sheathing parts made of aluminum alloy sheets are widely used in rocket and aircraft construction. The relatively low specific strength and corrosion resistance attracts designers, but the high cost and problems with achieving the necessary technological characteristics of shaping sheet material by wrapping in the manufacture of shells of complex configuration, limits the use of sheets of aluminum alloys with improved chemical composition due to microalloying with rare earth metals.

The characteristics of the shaping of the sheet material by the wrap in the manufacture of shells of complex configuration are determined by the changes in the material composition of the material, the level of hardening during plastic deformation and the anisotropy of the properties obtained during the tensile test of the samples, depending on the crystallographic texture of the sheet material. When the sheets are rolled and the shell parts are formed by the wrapping, heat is generated from them as a result of the dissipation of a part of the energy of mechanical action on the workpiece in these processes, performed at room temperature. This contributes to plastic deformation of the sheet blanks, which until now has not been considered a possible effect.

It is necessary to know the dynamics of heat perception of sheet material; its registration requires a higher measurement accuracy. It is possible to implement a variant of such a study when studying by methods of differential thermal analysis - DTA, with processing by methods of discrete analysis of data obtained by isothermal discrete scanning - IDS. There is already experience in rolling lithium alloys, when the thermal effects of deformation were taken into account [1].

The aim of the research is to show how the thermal effects during plastic deformation can be taken into account by the methods of temperature analysis. The object of the study was a sheet material of an aluminum alloy of grade 1163, which belongs to the class of duralumin with a very low content of impurities.
2. Experimental technique

Typical, as in heat treatment, the use of differential thermal analysis involves a continuous change in external temperatures. If it is combined with a gap in each act of iso-temperature discrete scanning, then the obtained data in the complex represent the changes occurring in the alloys in the form of distribution functions.

The possibilities of the method of isothermal discrete scanning are in expanding the application of temperature analysis for the study of structures [2]. Due to special heat treatment, it is possible to successively change not only the mechanism of dissipation of a part of the energy of mechanical action on the workpiece, but also to influence changes in the internal boundaries of the material composition of the alloy with control over the change in the sample volume during heating [3].

The change in the crystallographic texture of the material is accompanied by heat release. The properties of the subsequent resistance to corrosion cracking, intergranular and exfoliating corrosion depend on the cooling rate during quenching, technological background and sheet thickness. This is what is observed with DTA, while both processes and the movement of heat fluxes can be controlled with one device.

The choice of sheet material for research is the 1163 RDMV alloy of the Al-Cu-Mg system of regulated thickness and annealed is considered preferable as one of the main structural metallic materials in aircraft construction. Compared to other alloys of this system, it has increased strength, endurance, and higher fracture toughness, due to which it is actively used in products requiring a long service life in elements of aircraft structures. Alloy 1163 has a high level of fracture toughness and endurance characteristics; it is used in a naturally aged state (mode T) and after thermomechanical treatment (mode T7) [4]. But its working temperatures are only up to 80 °C, which is insufficient, and since sheets of alloys D16-T, D16ch.-T and 1163-T have a reduced corrosion resistance, the problem of their heat perception requires a solution.

Samples were taken from the delivery lot of OJSC "Kamensk-Uralsky Metallurgical Plant". Plated sheets of regulated thickness - soft, cladding. The mechanical characteristics of this alloy are given in GOST 21631-76, TU 1-801-84-83. For heating, 10 samples were made in the form of plates 1.8 mm thick, 250 * 20 mm in size. Pieces were cut into squares 5 * 5 mm, marked, subjected to discrete heating, scanned (continuous heating) and sent to storage in a dry desiccator. The thermograms were taken on a DTA-500 of the Trunin-Meshalkin scheme, a furnace designed by V.P. Egunov. The required modes of isothermal scanning were achieved by multiple holding of the samples separately.

In a laboratory oven in the thermostat mode at a given temperature, one sample of 1163 alloy of the same size with a set heating step was placed one by one. After holding, the samples were quickly removed from the oven chamber. The oven temperature is then raised to the next value. Heating cycles are repeated until the entire diagram is plotted, keeping constant: sample size, weight and holding time.

After measurements, each sample was subjected to isothermal holding for 1 minute in a DTA-500 at temperatures of 25, 171, 343, and 514 °C [3], cooled in air was held for the second time by continuous heating in the range of 100-550 °C, followed by cooling in the furnace chamber to 100 °C, then withdrawn for testing and characterization.

Preliminary processing of the results included the construction of a diagram of the change in the heating rate of the samples during scanning. The vertical axis is the heating rate, the horizontal axis is the continuous heating temperature. The curves represent the temperature of isothermal annealing: 171, 25, 343 and 514 °C. A sharp rise and a saddle in the region of 150 °C reflects the start of heating, then the rate of temperature rise increases in small steps, and short displacements of internal thermal processes occur in the sample. The lines show all the stages of

3. Processing of the obtained results of alloy 1163

Figure 1 shows the change in the heating rate of the samples during scanning. The vertical axis is the heating rate, the horizontal axis is the continuous heating temperature. The curves represent the temperature of isothermal annealing: 171, 25, 343 and 514 °C. A sharp rise and a saddle in the region of 150 °C reflects the start of heating, then the rate of temperature rise increases in small steps, and short displacements of internal thermal processes occur in the sample. The lines show all the stages of
what happens to the sample. Differences in the position of the lines relative to each other are associated with the processes of absorption and release of heat due to differences in isothermal holding. For specimens heated at a higher temperature (343, 514 °C), there is also a higher start at startup (more by 5 °C) and a higher heating rate and a continuing increase in the heating rate. From 425 °C, the steps in all graphs increase in size and, accordingly, their frequency decreases, the curve of the 514 °C sample differs from all the others, starting to separate at 343 °C.

![Figure 1](image)

**Figure 1.** Diagram of the change in the heating rate of samples of alloy 1163; Tia - isothermal annealing temperature, Tscan - continuous heating temperature.

Of particular note is the running of lines with a change in relative position. The most important factor in T^4^A (Temperature Analysis) analysis is a sign of a change in states. Despite the similar general nature of the lines, when viewed in a section diagram, they diverge, showing additional inflections - they are the desired ones. The temperature of preliminary holding (Tia) affects, the curves of 171, 25, 514, 343 °C are not arranged in order. In another section, there may be a different sequence. In order to see this, a special technique for representing images has been developed [5]. This is especially required when analyzing response lines during cooling.

Figure 2 shows the change in the cooling rate of the samples, which according to the indicated curves begins in the field of positive values - heating continues even when the furnace is turned off, which is associated with the inertia of the heaters. The heating rate decreases rapidly, turning into cooling - a field of negative values, thermal analysis recommends keeping in mind the boundary temperature of 514.5 °C. In contrast to thermal processes during heating, during cooling, there is no noticeable difference in the nature and quantitative parameters of the curves. It is the modes of cooling in thermography that are recommended for the diagnosis of chemical processes [6]. Here, the same sequence of thermal analysis - overlapping and overlapping does not mean data coincidence.
Figure 2. Diagram of the change in the cooling rate of isothermally kept samples of alloy 1163; $T_{ia}$ is the isothermal annealing temperature, $T_{scan}$ is the continuous cooling temperature.

Figure 3 shows the effect of the temperature of isothermal holding on the nature of the reactions of the samples under continuous thermal exposure. Stationary temperatures of 171.5 °C and 514.5 °C delimit heat perception in the sample, representing it in two separate states, up to 171.5 °C and after 514.5 °C. Stationary temperatures of 171.5 °C and 514.5 °C delimit heat perception in the sample, representing it in two separate states, up to 171.5 °C and after 514.5 °C. The temperature for 343 °C is considered as characterizing the force changes in the middle of the interval 250-343 °C of line 514, and two domes 250 °C and 450 °C, minima 171.5 °C and 514 °C. This layout shows the relationship with the redistribution of graininess in the volume.

Figure 3. Fragment of heating 1163, the effect of isothermal exposure on the difference in electrical potential during differential thermal scanning - TTS; $T_{ia}$ - isothermal heating temperature, $T_{scan}$ - continuous cooling temperature.
The nature of the lines below 171.5 °C shows the thermal dynamics of the entire sample. Above 514.5 °C, fragmented melting already begins, with holding for one minute there is not enough energy to cover the entire volume of the sample during this time. At 343 °C, small anomalies and orthogonality of the transition, processes at the contact of crystals are noticeable. According to the temperature analysis, the processes occurring at 514.5 °C are characterized by the transformation of the stationarity of the regime in the volume of the substance [7, 8].

On figure 4 it can be seen how the emphasis shifts in the character of the curve near the stationary temperature of 514.5 °C, which is especially noticeable for the sample processed at 514 °C. At the same time, if for treatments 25, 171 and 343 °C before 514.5 °C there appears a step with a sharp increase in Δthermo EMF, then after treatment 514 °C this is a saddle before lifting. The heat transfer area is expanded - this is a sign of structural changes.

Figure 4. Comparison of the 450-550 °C section from Figure 3 with the 514.5 °C point (half-interval of stationary temperature) and without it. 25, 171, 343 and 514 in the legend - designations of the temperature of isothermal heating; Tscan is the continuous cooling temperature.

Figure 5 shows the cross-sections of the heating diagrams of the samples by the temperatures of continuous heating. The curves of 100 and 525 °C stand out especially - they can be used to determine the boundary of the contact surface.
Figure 5. Diagram of the influence of the isothermal holding temperature on the difference in the electrical potential of the sample during heating; curves - temperature of continuous heating. The horizontal axis of temperatures of isothermal heating (Tia) axis.

Figure 6 shows an analogue of the 3D model combining figure 3 and figure 4. Peaks at 343 and 514 °C at the beginning of heating are clearly shown, and peaks at the end of heating at 25, 171 and 343 °C. Curve 343 °C has a high peak at the beginning of heating and at the end, while it does not have a "pit" in the middle of the range. A more convenient version of a histogram for viewing data in a three-dimensional graph is a surface (Figure 6). The advantage of such histograms is the ability to accurately determine the parameters of the secondary scan.

Figure 6. Fragment of heating, histogram of the effect of the temperature of continuous heating on the difference in the electrical potential of the sample during isothermal holding; Tia is the isothermal heating temperature, Tscan is the continuous cooling temperature.
Figure 7. Fragment of heating, histogram-surface of the influence of the temperature of continuous heating on the difference in the electrical potential of the sample during isothermal holding; Tia is the isothermal heating temperature, Tscan is the continuous cooling temperature.

In figure 7 that an increase in the iso-holding temperature increases ΔthermoEMF, being especially pronounced at 100 and 550 °C (high peaks), reaching a general maximum at 343 °C and decreasing at 514 °C, except for the highest peak at 100 °C. The diagram in figure 7 according to TMA very clearly reflects the general tendency of transformation to the stationarity temperature.

Figure 8. Influence of isothermal exposure on the difference in the electrical potential of the sample during thermal scanning for 1163; fragment - cooling; Tscan is the continuous cooling temperature.
On figure 8, the cooling for all curves has a uniform character, steps are visible at 400-450 and 250-275 °C. The peculiarity of cooling is that after 125 °C the electric potential of the samples grows and is restored by 40-45% of the initial one at the beginning of cooling. And these lines clearly show the nature of the accuracy of the work of the IDS method when optimizing the modes - the lines strictly follow their own trajectories, the states are characterized as steady.

![Figure 8](image)

**Figure 9.** Diagram of the effect of the temperature of isothermal holding on the difference in the electrical potential of the sample during cooling; curves - temperature of continuous heating. The horizontal axis of temperatures of isothermal heating (Tia) axis.

Figure 9 shows the cross sections of the cooling curves of the samples. The curves are smooth and homogeneous. The lowest levels on the Δ-axis are thermoEMF at temperatures of 125 and 150 °C. The 550 °C curve has the highest ΔthermoEMF. The highest density of curves is in the region of zero difference in electric potentials.

![Figure 10](image)

**Figure 10.** Fragment of cooling in the form of a histogram of the effect of the temperature of continuous heating on the difference in the electrical potential of the sample during isothermal holding; Tia is the isothermal heating temperature, Tscan is the continuous cooling temperature.
Upon cooling, the electrical potential of the samples passes into the field of negative values with a tendency to recover in the field of positive values upon further cooling after 125 °C.

Figure 11. Histogram-surface of the influence of the temperature of continuous heating on the difference in the electrical potential of the sample during isothermal holding for 1163; fragment - cooling. Tia is the isothermal heating temperature, Tscan is the continuous cooling temperature.

Figure 11 shows the cooling of samples previously discretely fired at 25, 171, 343 and 514 °C and then continuously heated to 550 °C. The nature of the cooling is uniform, the processing temperature has almost no effect on the internal processes during the cooling of the sample. Two steps and a saddle are clearly visible, after which the electropotential of the sample tends to recover upon cooling to less than 125 °C. Key temperature 171.5 °C.

4. Summary
The processes of heat perception in the samples during heating and cooling have characteristic effects at 171, 343, 514 °C.

A sharp increase in the heating rate at the beginning, then a decrease, the formation of a “saddle” and then a linear increase in the rate shows that the heat perception of the sample refers only to the outer part of its volume, has a layer that has established itself in temperature, and then continues along the classical gradient. Such data can be scaled by reference to the dimensions of the sample. This also means the parameters of plastic deformation.

The difference in thermal processes in the dynamics of heating and cooling is associated with the redistribution of grains by size. Upon cooling, there is no noticeable difference in the nature of structural changes, the heating is completed, and the heat perception is leveled, in fact, the material has become more homogeneous.

The DTA data showed that the greatest dynamics of changes in the heat perception of alloy 1163 occurs during processing at 514 °C.

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