

Establishment of continuous cooling transformation diagrams of aluminum alloys using in situ voltage measurement

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Abstract: An effective method was proposed to establish the continuous cooling transformation (CCT) diagrams of aluminum alloys using in situ voltage measurement. The voltage change of samples with predefined dimension was recorded under the constant current state during continuous cooling. Solutionizing time, together with starting and finishing temperatures of phase transformation of the alloy can be obtained from relationships of voltage vs time and temperature. A critical cooling rate without detectable phase transition during continuous cooling can be determined. Continuous cooling transformation diagrams of tested samples can be established conveniently based on these results. Microstructure observation and differential scanning calorimetry (DSC) testing were applied to verify the reliability of continuous cooling transformation diagram.

Key words: aluminum alloys; in situ voltage measurement; phase transformation; CCT diagram; microstructure

1 Introduction

Quenching is one of the key issues to affect the properties of aluminum alloys [1–3]. With a full continuous cooling transformation (CCT) diagram, it is possible to get the optimal quenching rate of an aluminum alloy. Method of establishing CCT diagrams of steels is a mature technique, and many CCT diagrams of steels have been widely studied and used in industry [4–5]. However, conventional procedures of producing CCT diagrams of steels, such as expansion method, are not suitable to establish the CCT diagrams of aluminum alloys. Fortunately, thermal analysis, especially differential scanning calorimetry (DSC) provides an approach for CCT diagrams of aluminium alloys [6–7]. However, DSC method has its limits due to its narrow cooling range, which cannot reach the critical quenching rates of some aluminum alloys.

Electrical resistance of a material is sensitive to the change of its microstructures [8–9]. It varies obviously during phase transformation, such as clustering of atoms and dissolving of precipitation. Therefore, attempts to characterize the precipitation processes in aluminium alloys often involve electrical resistance measurements [10–12]. In our previous research, the CCT diagram of

Al-Cu-Li alloy was determined using relative resistivity measurements in parallel with DSC analysis and microstructure observation [13]. However, the determined CCT diagram still needs to be improved because the phase transformation temperature cannot be judged accurately. The aim of this work is to find a more effective approach to establish CCT diagrams of aluminum alloys. In the present study, in situ voltage measurement of sample under a constant current state to establish CCT diagrams of aluminum alloys is put forward, which can be conveniently used to characterize the phase transformation behavior.

2 Experimental

The investigated alloy was a forged 7075 alloy whose composition is given in Table 1. Samples for testing were cut into slender rectangles by spark machining. A voltage and temperature testing system was designed [14].

Table 1 Chemical composition of investigated 7075 alloy (mass fraction, %)

Mg	Zn	Cu	Si	Fe	Mn	Cr	Ti	Ni	Al
2.55	5.44	1.37	0.15	0.39	0.22	0.21	0.05	0.02	Bal.

The solutionizing temperature was ascertained according to overburning temperatures measured by DSC. A constant current was applied to heat the samples from room temperature to solutionizing temperature. The voltage variation with the heating time was recorded, from which the solutionizing time can be selected. In order to get different quenching rates, the samples were cooled in various medium (furnace cooling, air, wind and liquid nitrogen). The voltage variation with the temperature was measured. The average cooling rate can be calculated from the cooling curves. The starting and finishing phase transformation temperatures and the critical cooling rate without phase transition can be ascertained based on the slope of voltage—temperature curves. CCT diagram can be plotted accordingly.

The samples were quenched with the same cooling rate from five different temperatures (A: solutionizing temperature; B: above the starting temperature of phase transformation; C: a little below the starting temperature of phase transformation; D: in the middle of the starting and finishing temperatures of phase transformation; E: below the finishing phase transformation temperature). Their microstructures were observed using transmission electron microscopy (TEM) to verify the phase transformation temperatures. Samples for TEM observation were prepared by twin jet-polishing using a 10% (volume fraction) solution of nitric acid in methanol. TEM observations were performed with a TecnaiG²20 ST microscope operating at an accelerating voltage of 160 kV.

3 Results and discussion

Figure 1 shows the DSC curves of the tested alloy heated at the rates of 5, 10 and 15 °C/s, respectively. The peak values which represent overburning temperatures were all about 485 °C. The solutionizing temperature of 7075 alloy should be below 485 °C, so 475 °C was

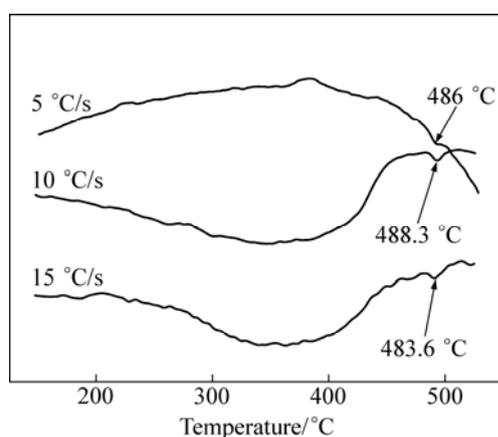


Fig. 1 DSC curves of 7075 alloy with heating rates of 5, 10 and 15 °C/s

selected as the solutionizing temperature. Figure 2 shows the relationship between voltage and time when the sample was held at 475 °C. The voltage increased with time rapidly within the initial 30 min, while it increased slowly thereafter. After 60 min, the voltage kept constant with increasing time, indicating that the samples were fully solution treated. So (475 °C, 1 h) was selected as the solution process for the tested alloy.

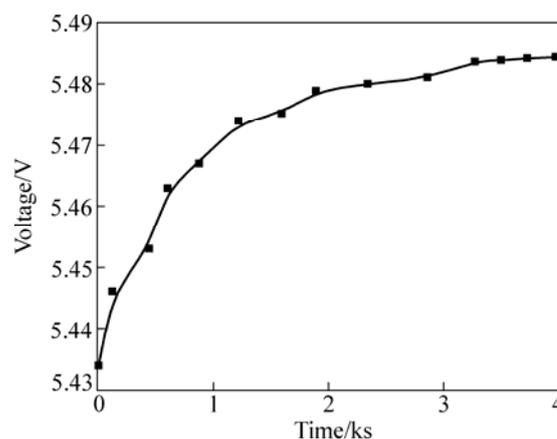


Fig. 2 Voltage versus time curve of tested alloy when solutionizing at 475 °C

The typical voltage versus temperature curves for 7075 alloy with different cooling rates are presented in Fig. 3. The voltage versus temperature curve is composed of three sections with different slopes at a cooling rate of 0.030 °C/s (Fig. 3(a)). According to Matthiessen rule, the resistance of solution treated alloy declines linearly with decreasing temperature. The voltage versus temperature curve displays a straight line as phase transformation does not take place during continuous cooling. Once solute atoms precipitate from solid solution, and precipitation of new phase occurs, voltage versus temperature curve deviates from straight line, which is shown at the temperature between 208 °C and 417 °C (Fig. 3(a)). The voltage versus temperature curve returns to straight line again as the transformation finishes [15]. As the cooling rate is lower than 0.251 °C/s, the voltage versus temperature curves are similar to the curve shown in Fig. 3(a).

It is noteworthy that the voltage versus temperature curves are composed of two straight-line segments with the cooling rate faster than 3.443 °C/s. As the samples are cooled to room temperature at a cooling rate of 9.465 °C/s, the voltage versus temperature curve begins to deviate from the original straight line at 328 °C (Fig. 3(b)), and no definite finishing temperature of the transformation appears. When the samples are quenched at a high cooling rate, the solute atoms do not have enough time to precipitate from the solid solution, resulting in a high super-saturated solid solution. Phase

transformation continues at room temperature due to the high driving force of phase transformation. Hereby, phase transformation temperatures of 7075 alloy at various cooling rates are summarized and listed in Table 2.

The curve shown in Fig. 3(c) is approximately a straight line in the whole temperature range. It illustrates that the cooling rate has already approached or exceeded the critical cooling rate. The cooling rate corresponding to the curve shown in Fig. 3(c) is higher than that in Fig. 3(b), however, the curve in Fig. 3(b) is composed of

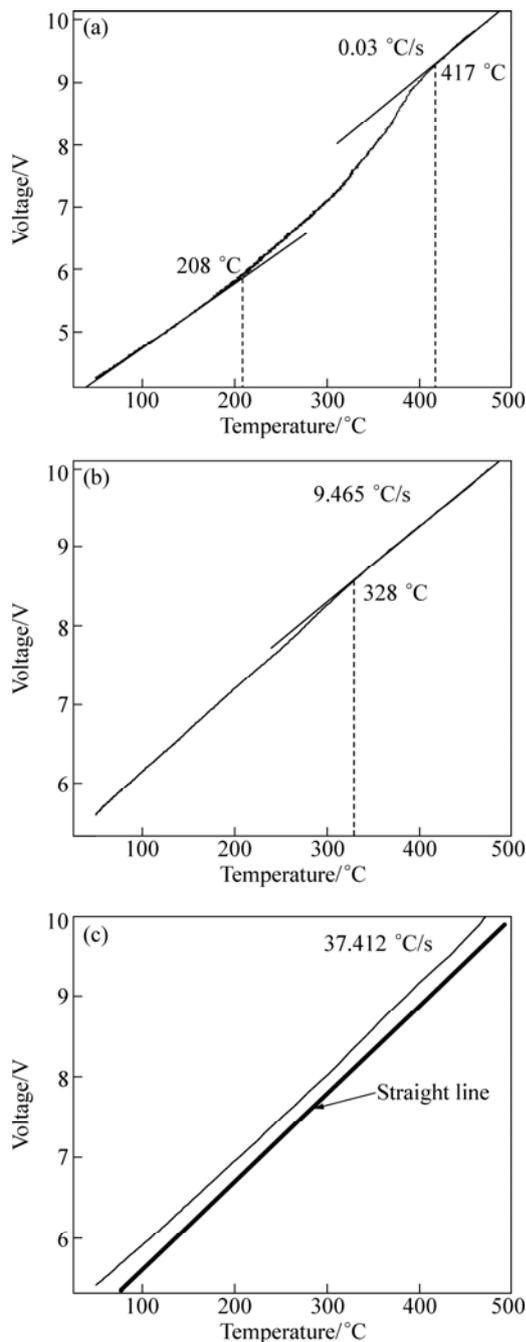


Fig. 3 Voltage as function of temperature during continuous cooling at different quenching rates: (a) 0.030 °C/s; (b) 9.465 °C/s; (c) 37.412 °C/s

Table 2 Phase transformation temperatures of 7075 aluminum alloy at various cooling rates

No.	Average cooling rate/(°C·s ⁻¹)	Start temperature/°C	Finish temperature/°C
1	0.008	430	214
2	0.030	417	208
3	0.042	408	200
4	0.060	405	195
5	0.251	398	171
6	3.443	367	–
7	4.798	365	–
8	9.465	328	–
9	37.412	–	–

two straight line segments with different slopes, indicating that this cooling rate does not approach the critical one, under which the phase transformation can be inhibited. Therefore, the critical cooling rate should be located between 9.5 and 37.4 °C/s.

The curves at different cooling rates are plotted in one diagram (Fig. 4). The time-axis is scaled using logarithmic. Starting and finishing temperatures of phase transformation under the specific cooling rate are marked at corresponding cooling curves and connected by smooth line respectively. Solutionizing temperature and time, together with critical cooling rate are also marked. The constructed CCT diagram is presented in Fig. 4, in which phase β includes all possible secondary phases, such as MgZn₂ and Al₂CuMg. According to Fig. 4, phase transformation takes place mainly in intermediate temperature range (170–430 °C). Interrupted quenching process can be determined according to the established CCT diagram, so it is possible to decrease the quenching stress and to inhibit decomposition of solid solution.

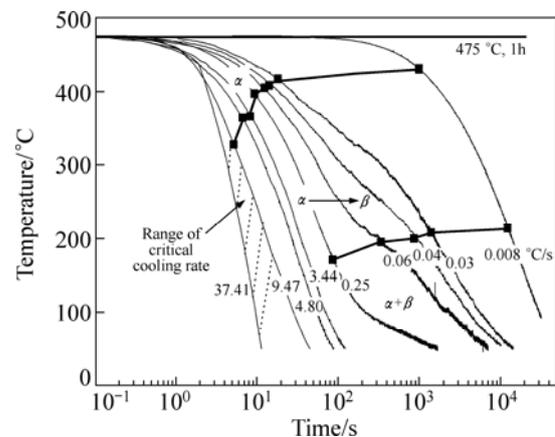


Fig. 4 CCT diagram for 7075 alloy

Samples were cooled at a cooling rate of 0.03 °C/s and quenched from 475, 425, 410, 300 and 50 °C, respectively. Figure 5(a) shows the microstructure of the forged sample without solution treatment. Microstructure

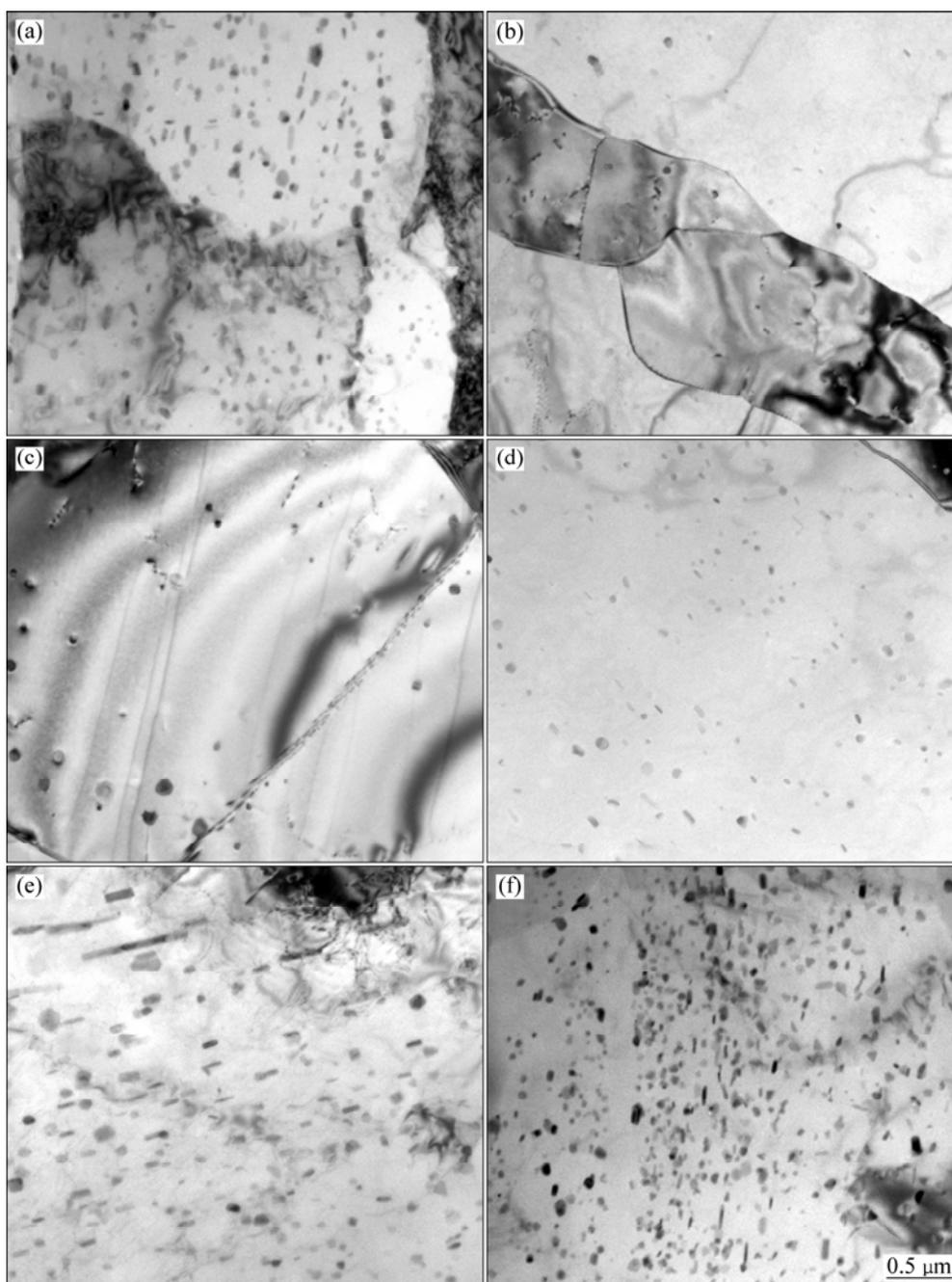


Fig. 5 TEM bright field images of samples without solution treatment (a) and quenched from 475 °C (b), 425 °C (c), 410 °C (d), 300 °C (e) and 50 °C (f)

evolutions of 7075 alloy during cooling are shown in Figs. 5(b–f). It can be observed that a great deal of precipitations and dislocation tangle in the forged alloy (Fig. 5(a)). However, after solution treatment, there are only a few precipitations (Fig. 5(b)). As the sample is quenched from 410 °C, some secondary phases precipitate. The number and dimension of the secondary phases increase with the decreasing of quenching temperature, as shown in Figs. 5(c–f). The nucleation, coarsening and increasing of precipitations result in the slope variations of the voltage versus temperature curve. TEM observation proved that using voltage versus

temperature curves to determine the transformation temperature is available and credible.

The temperature versus time curve corresponding to Fig. 3(a) is showed in Fig. 6. The section of the curve between 300–470 °C is an approximate straight line, and the average cooling rate corresponding to this section is 0.506 °C/s. The sample was held at 475 °C for 1 h prior to DSC measurement, and then cooled to 300 °C at the cooling rate of 0.506 °C/s. The DSC curve and its derivation on time are shown in Fig. 7. A peak at 421.6 °C can be seen from the derivation curve, which illustrates that the precipitations have occurred. The

starting transformation temperature can be measured using in situ voltage measurement (Fig. 3(a)). The difference of the measured result using the two methods is only 4.6 °C. Considering the experimental errors and the difference between practical cooling and linear continuous cooling, the starting transformation temperature measured by voltage versus temperature curves coincides well with that measured by DSC. Both TEM observation and DSC testing proved that using voltage versus temperature curves to determine the transformation temperature is available and credible.

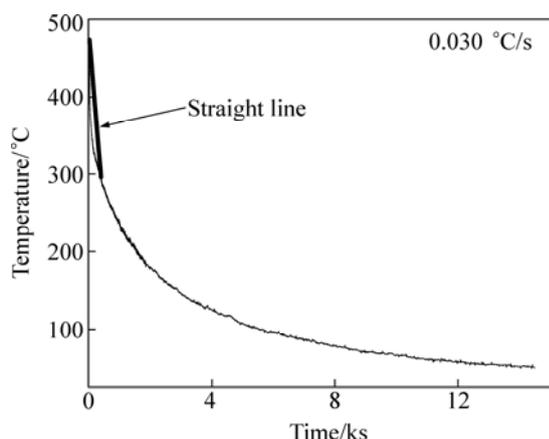


Fig. 6 Temperature vs time curve corresponding to Fig. 3(a)

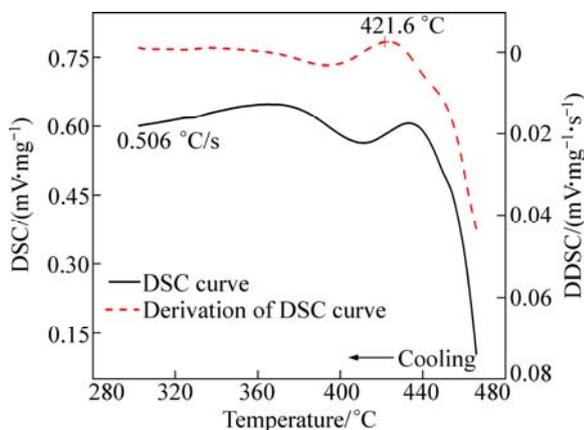


Fig. 7 DSC curve and its derivation curve of 7075 alloy

The CCT diagrams of aluminum alloys were successfully established using in situ voltage measurement. Traditional in situ electrical resistance measurement was replaced by magnified in situ voltage measurement, which can effectively overcome the problem that the resistance of aluminum alloy is too small to exactly collect and measure during continuous cooling. The voltage variations can accurately reflect resistance evolution under the constant electric current. Using voltage versus time curve, appropriate solutionizing time can be gained, and the reasonable solutionizing process of aluminum alloys, especially for

new type ones, can be obtained effectively and economically. The starting and finishing temperatures can be easily gained through the slope change. However, the established CCT diagram has a few flaws. For example, the specific kinds of precipitated phases cannot be indicated in the diagram. It will be one of the tasks of future work.

4 Conclusions

1) A novel method was successfully put forward to establish the CCT diagrams for aluminum alloys using in situ voltage measurement. Magnified in situ voltage measurement can accurately reflect microstructure evolutions of aluminum alloy during continuous cooling.

2) The reasonable solutionizing time, together with starting and finishing temperatures of phase transformation and the critical cooling rate of the tested alloy can be ascertained based on the relationships between voltage and time, voltage and temperature. Therefore, CCT diagrams can be plotted.

3) Both TEM observation and DSC testing verified the reliability of transformation temperatures, which were obtained from voltage versus temperature curves. It is a convenient and accurate method for the establishing of CCT curves of aluminum alloys using in situ voltage measurement.

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用原位电压法测定铝合金的 CCT 图

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摘要: 提出一种用原位电压法测定铝合金连续冷却转变图(CCT图)的方法。采用自主开发的设备,在恒电场作用下,测试一定尺寸的试样在保温及连续冷却过程中的电压变化曲线。根据电压—时间及电压—温度曲线,获得实验合金的最佳固溶时间、相变开始温度和相变结束温度,并确定实验合金抑制相变发生的临界淬火速率范围。根据相变信息建立实验合金的CCT图,采用透射电镜观察(TEM)和差示扫描量热(DSC)测试验证了CCT图的准确性。

关键词: 铝合金; 原位电压法; 相变; CCT图; 微观组织

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