PHASE SEPARATION PROCESSES AT EARLY STAGE OF AGEING IN ZA27 ALLOY①

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ABSTRACT Phase separation processes of cast ZA27 alloy aged at room temperature and 100 ℃, respectively were studied by means of transmission electron microscope, X-ray diffraction and microhardness measurment. It was concluded that G P zones form at early stage of ageing phase separation when ZA27 alloy is aged at room temperature, but the alloy undergoes spinodal decomposition process, in which concentration waves develop along ⟨111⟩, a soft direction in elastic, when aged at 100 ℃.

Key words Zn-Al alloy spinodal decomposition G P zone phase transformation

1 INTRODUCTION

Cast Zn-Al alloys are a series of newly developed structural materials[1], including ZA8, ZA12 and ZA27, which are strengthened during aged process due to a continuous phase separation process in which there are some uncertainities by now. Some researchers[2-4] suggested that the process proceeds as follows: $\alpha_{SS} \rightarrow$ coherent global G P zones → elliptic G P zones → stable phases. On the contrary, Schwarh *et al*[5], Vilayalakshmi *et al*[6] and Ungar *et al*[7] interpreted the phase separation at early stage of ageing according to spinodal decomposition (SD) mechanism on the basis of experimental results of XRD, TEM and DSC. Yao *et al*[8] considered that it was possible for the formation of G P zones and SD to occur simultaneously upon various ageing stages. In present work, a series of experiments with TEM and XRD have been conducted to distinguish phase separation modes during ageing in ZA27 alloy.

2 EXPERIMENTAL

The ZA27 alloy used in the study was melted in a graphite crucible with pure zinc (No. 1) and A-00 aluminium. An H-800 TEM equipped with a high angle, double-tilt metallurgical stage, a Rigaku X-ray, diffractometer equipped with copper target and Ni filter and a Micromet' II microhardness tester were used in the investigation. The TEM foils were prepared by jet electropolishing with an electrolytic solution of 10% (in volume) perchloric acid plus alcohol.

3 RESULTS AND DISCUSSION

The ZA27 alloy in the study was first annealed at 400 ℃ for 5 h and solution-treated at 390 ℃ for 1 h, then aged at room temperature (RT) and 100 ℃, respectively, for various time. Fig. 1 shows the micro-hardness changes with aged time. Both curves can be divided into three stages: hardness rising slowly, peak hardness and hardness decreasing, though each stage took various aged time at different aged temperatures. The hardness changing with aged time at RT is slower than that of at 100 ℃, for example, the hardness of samples aged at RT for 20 h is equal to that of aged at 100 ℃ for 5 min. It took about 60 h for the hardness of a sample aged at RT to reach its peak hardness, which is much longer than that time needed for aged at 100 ℃. And we can also find that there is a hardness plat stage from 5 min to 3 h in the curve of aging at

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100 ℃.

The XRD patterns of specimens aged at RT for various time are presented in Fig. 2.

To avoid phase separation during solution quenched and enhance the comparability of various patterns aged for various time , the method of quenching specimens in the ice brine water and moving it into liquid nitrogen immediately was needed. The specimens in liquid nitrogen were taken out till it' s time for XRD analysis. They were fixed at the same spot of XRD instrument. This would be done after specimens were aged for various time. That the diffraction pattern of as-quenched alloy as shown in Fig. 2 indicates the formation of stabilized η phase (zinc-rich phase) shows that the quenching rate is too slow to prevent the phase decomposition completely . Fig. 2 also shows the amount of η phase precipatation is increased and the intensity of diffraction peak is risen with the aging time prolonged. Because no sidebands or satellite peaks are found around the (200) or (111) main diffraction peak for different aging time, it suggests that SD may not take place during the natural aging process. The conclusion is affirmed by TEM investigation. Fig. 3 shows the morphology of specimen aged at RT for 15h observed by TEM, G P zones which is about 200 Å in diameter dispersed in the matrix, but no characteristic structures of SD, i. e. wavy contrast and modulated structure are found in it. TEM observation showed that there were wavy contrast taking on in the matrix of specimen aged at 100 ℃ for 2 min (Fig. 4(a)) and correspondingly the diffraction patterns were stretched in ⟨111⟩ orientation (Fig. 4(b)) along with the solute-rich area and solute-depleted area are probably formed. We can concluded that SD has taken place during aging of supersaturated solution of ZA27 alloy at

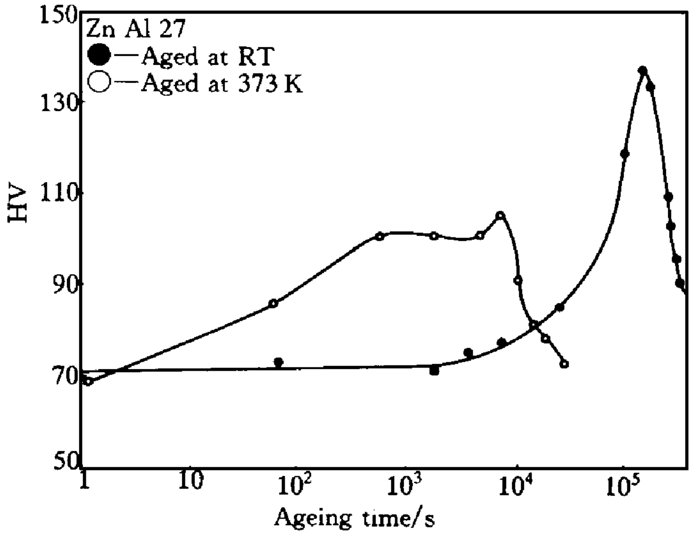

Fig. 1 Microhardness variations of cast ZA27 alloy aged at RT and 100 ℃

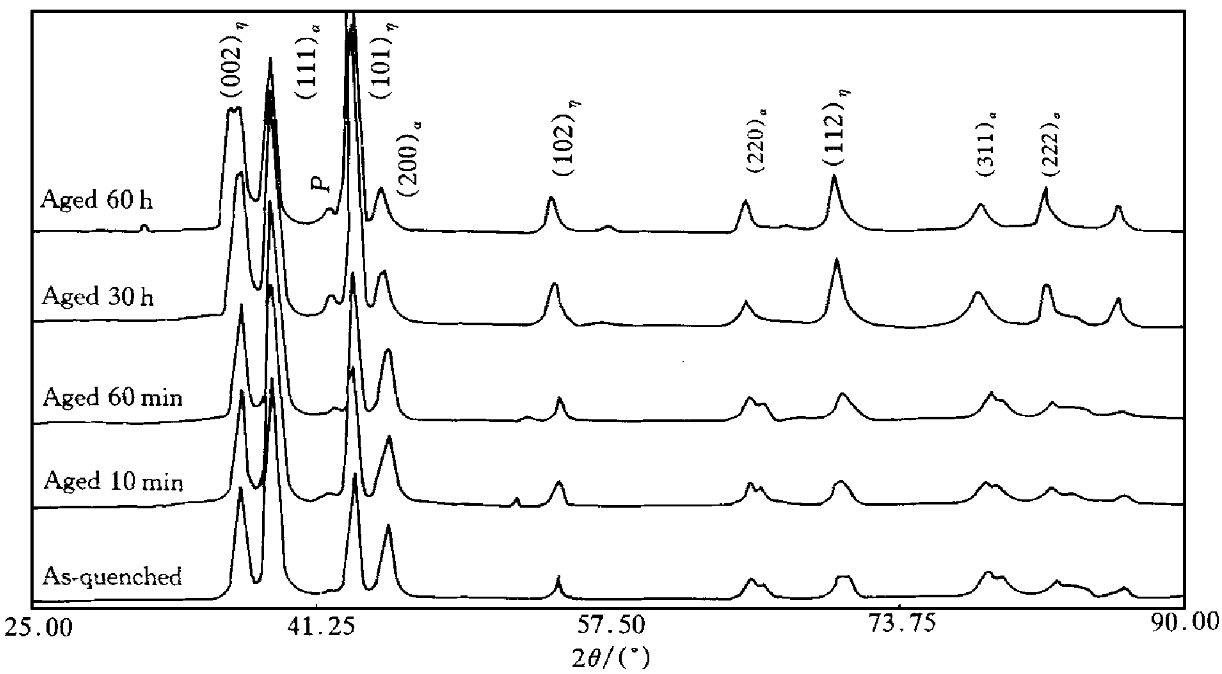

Fig. 2 XRD patterns of specimens aged at RT for various times

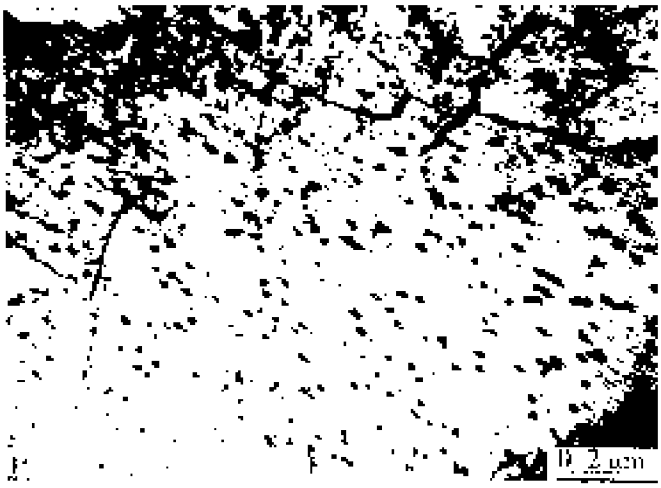

Fig. 3 TEM photograph of cast ZA27 alloy aged at RT for 15 h

100 ℃. Modulation wavelength, λ is about 80 Å when measured in microstructure. The wavy contrast developed into modulated structure (MS) after aged at 100 ℃ for 30 min while the λ is about 500 Å

SD taking place during aging process belongs to continuous phase transformation. Its criterions are generally wavy contrast of the matrix, XRD sideband and satellite in diffraction pattern, and so on. In the investiagtion of aging process of ZA alloy, many researchers took MS as the criterion of SD, it is unreasonable because nucleation-growth can also result in MS. In the meantime, SD can develop many other structures such as island structure formed in Fe-Cr-Co aged alloy. However, the wavy contrast, which is a kind of strain in the matrix caused by solute atoms aggregating along soft elastic direction during SD, is the sufficency condition for SD. With the wavy contrast observed in ZA27 alloy aged at 100 ℃ as well as the fact that main electron diffraction patterns have been stretched, we can conclude that ZA27 alloy in the state decomposes through SD mechanism unambiguously.

The content of Al atoms in ZA27 alloy is up to 48%, which is nearly the same as that of Zn, so there may be some difficulties to determine the soft elastic direction along which SD takes place. For Al, the fcc lattice, its soft elastic direction is ⟨100⟩, but that of Zn is uncertain because zinc belongs to the hcp lattice. As Fig. 2 indicates, η phase has precipitated in as quenched and the amount increased with the

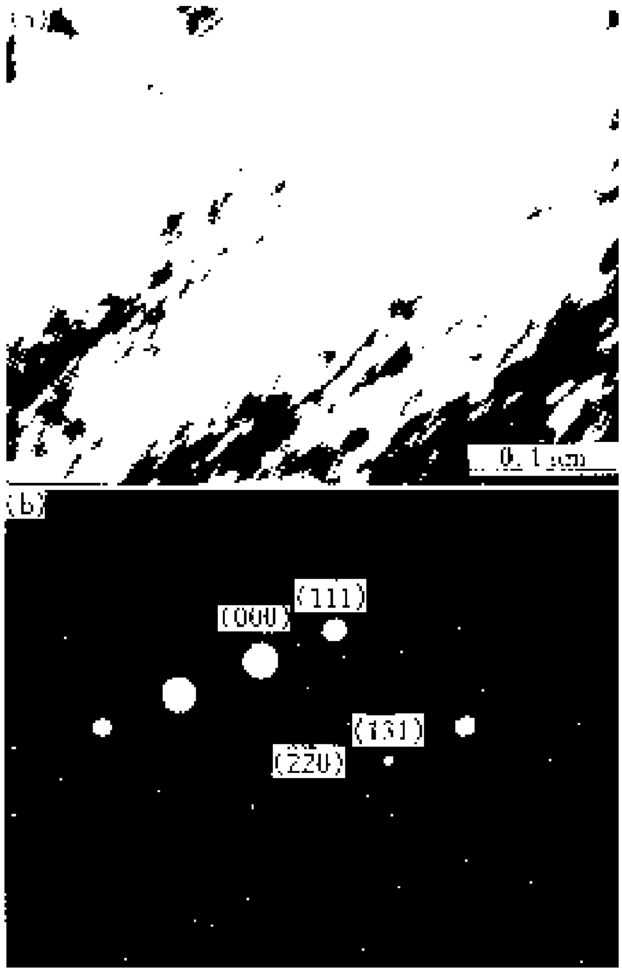

Fig. 4 Wavy contrast of ZA27 alloy aged at 100 ℃ for 2 min

(a) —wavy contrast due to SD;
(b) —electron diffraction pattern in selected area

Fig. 5 Modulated structure of ZA27 alloy aged at 100 ℃ for 30 min

aging time prolonged. It can be infered that the matrix of ZA27 alloy may be Al-riched solution in which SD took place along the ⟨100⟩ soft elastic direction. Because of the relatively small elastic anisotropic factor for Al[12], ⟨111⟩direction may also become an aggregated orientation of sulute atoms. This inference is affirmed by the direction along which wavy contrast appeared and the diffraction pattern which was stretched in ⟨111⟩ direction as shown in Fig. 4. References [8] and [11] suggested that the soft elastic direction of ZA27 alloy is ⟨111⟩. But reference[6] suggested that the soft elastic direction is ⟨100⟩ by studying of relatively low Zn containing alloys' phase decomposition. This difference may be related to the different Zn content in the studied alloys.

Now let's reconsider the changes of microhardness of specimens aged at RT and 100 ℃. In the latter case the hardness rose gradually within very short time and formed a flat stage. This was the result of SD enhanced by larger heat activation process. At the early stage of SD the hardness rose rapidly due to strong strengthening effect to the matix. After the wavelength of SD reached a certain degree, the hardness changed slowly with aging time and the strengthening effect decreased. Therefore, a flat stage was formed in hardness curves. With the aging time prolonged further, the MS became coarsen and the hardness decreased, while the alloy was aging at RT, its atoms moved slowly due to lower heat activation. As a result, the formation of G P zones needed a long period and its hardness rose slowly till it got to the peak with the converting of G P zone to a metastable phase. After that, the hardness would decrease with the formation of stabilized phase, which was called hyperaged. The fact that the peak hardness of alloy aged at 100 ℃ was lower than that of aged at RT may be a result of the cellular reaction[13] taking place in SD easily at high heat activation and with the aged accordingly.

4 CONCLUSIONS

The phase separation process of casted ZA27 alloy aged at 100 ℃ abides by the principle of SD and that of aged at RT by nucleation-growth and coarsening to form G P zone. The soft elastic direction of matrix of ZA27 alloy while it undergoes SD is along the ⟨111⟩ direction.

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