Microstructure Observation of Naturally Aged Thixoforming ZA27 Alloy

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The microstructure of the thixoformed ZA27 alloy naturally aged for 5 years has been observed and analyzed. The results indicate that most of the original β + η laminar eutectics have changed into the structures that small α particles uniformly distribute in η phase matrix. Both the β phase and α' phase have also decomposed. It is suggested that a continuous or dot-line α phase first precipitates in the β phase close to the eutectic η phase layer. Then eutectoid η phase nucleates or directly grows up from the eutectic η phase, which promotes the nucleation of eutectoid α phase or the direct growth of the α phase from the existing α phase. Subsequently, the resulting α + η phases grow towards the β phase inside, forming the regular laminar α + η structures. The laminar structures always continuously grow across the center α' phase, which leads the α' phase also to transform into regular α + η laminar structures. The laminar structures may crack possibly due to internal stress. The eutectoid η phase lamellae have a tendency of changing from laminar shape to particle form, resulting in the formation of non-laminar structures.

Keywords: *ZA27 alloy, thixoforming, ageing, microstructure*

1. Introduction

Zn-Al alloys containing small amount of copper have been observed to be cost- and energy-effective substitutes to a variety of ferrous and non-ferrous alloys owing to their higher strength, better wear and friction properties, lower casting temperature and more abundant resources[1,2]. Of the newer alloy, ZA27 is of particular interest because it has high values of tensile strength up to 450MPa at ambient temperature[3]. Unfortunately, it suffers from degradation of mechanical properties due to easy formation of porosity because of its wide solidification interval[4,5].

Thixoforming, however, a relatively novel metal-processing technique that combines the elements of both casting and forging, offers significant advantages and one of which is reducing or eliminating porosity[6-8]. The authors' previous investigation indicated that the tensile properties of thixoformed ZA27 alloy were significantly improved because of the obvious decrease of porosity, compared with those of the permanent mould cast alloy[9]. It is known that the microstructures of Zn-Al alloys are very complicated because their solidification process not only involves peritectic and eutectic reactions, but also a series of solid phase transformations[10]. The existing investigations have studied the microstructures of as-cast or aged Zn-Al alloys[10-13]. The results show that the as-cast microstructure of the ZA27 alloy can be well explained through analyzing its solidification process[10-13]. The solidification of the ZA27 alloy begins with the precipitation of Al-rich α' phase and thus developed α' dendrites first form. Then a peritectic reaction of L + α' → β occurs and a Zn-rich β phase layer is generated on the α' dendrite surface, forming a kind of shell-core structured dendrites. Finally,

the residual liquid solidifies through a eutectic reaction of L → η + β + ε (CuZn$_4$) and the ternary eutectic structures are formed between the peritectic β phases. But the aged microstructure, especially the relationship between the ageing process and the resulting microstructure is still unclear[10-13]. The confirmed result is that both the α' and β phases are unstable and will decompose into the equilibrium phases of α (a Al-rich fcc solid solution), η and ε. Furthermore, there is a large difference in microstructures between the traditionally as-cast and as-thixoforming alloys[14]. But no investigations on the microstructure of the aged thixoforming ZA27 alloy can be found. Therefore, the microstructure of a thixoformed ZA27 alloy naturally aged for 5 years has been characterized in this work. Especially, the formation process of the microstructure has been discussed. It can be expected that this investigation is helpful for the application of thixoforming ZA27 alloy.

2. Experimental Process

The nominal compositions of the used ZA27 alloy are 26-28% Al, 1.7-2.0% Cu, 0.2% Zr, 0.02-0.04% Mg and balance of Zn (in wt.%). The Zr element is used to refine the primary α'–Al grains so as to obtain a nondendritic semisolid microstructure after being partially remelted[9]. A quantity of pure Al, Zn, Mg and Al-50 Cu master alloy was melted at 670-700 °C and 0.2% Zr was then added in form of K$_2$F$_6$Zr. Subsequently the melt was degassed by C$_2$Cl$_6$ and cooled to 550 °C, and finally poured into a permanent mould with a cavity of Φ 45 mm × 190 mm. Chemical analysis shows that the actual compositions of the resulting alloy are 26.83% Al, 1.86% Cu, 0.17% Zr, 0.02% Mg and balance

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of Zn. The resulting casting rods were machined into small rods with dimensions of Φ 45 mm × 50 mm. One small rod was put into a resistance furnace with a temperature 475 °C and heated for 90 min to obtain a semisolid ingot. Then the ingot was handled into a mould chamber fixed on a pressure machine and thixoformed using a pressure of 40 ton. The employed mould temperature was at 300 °C. Repeating the experimental procedure, some thixoformed rods with dimensions of Φ 15 mm × 165 mm were obtained and then naturally aged for 5 years. The previous investigations indicated that the processing parameters used in this work were suitable for forming the ZA27 alloy and the resulting alloy had quite high tensile properties[9,15].

Some small metallographic specimens were cut from one rod, and finished and polished. Then they were carefully observed on a JSM-6700F scanning electron microscope (SEM) using back-scatted imaging technique and analyzed using energy dispersive spectrometer (EDS). Then the specimens were etched using a 4% HNO_3 aqueous solution and observed on a Mef-3 optical microscope (OM). To examine the phase constituents, one specimen was analyzed on a D/MAX-2400 X-ray diffractometer (XRD).

3. Results and Discussion

3.1. General microstructure

Figure 1a shows that the microstructure of the thixoformed ZA27 alloy is composed of near spheroidal primary particles and intergranular secondarily solidified structures. But the microstructure of the traditionally cast one is consisted of equiaxed dendrites and interdendritic eutectic structures[9]. Namely, the microstructures of the alloys prepared by these two forming technologies are very different. In addition, there are always several secondarily solidified pools with different sizes in each primary particle. And also a large-sized one always exists in the center of each particle, which leads the primary particles appearing in ring-like structures. The pool-like structures solidify from the liquid pools within the primary particles in semisolid state. The formation of the liquid pools has been detailedly discussed in references[16,17]. Figure 1b shows that there is a grey layer between the secondarily

solidified structures and the white primary particles. In the large-area regions among the primary particles, there are some individual small grey grains while in the small-area or thin regions, the primary particles are connected together by the grey layer structures.

To more obviously show the microstructure, the specimen has been observed by SEM as shown by Figure 2. Comparing Figures 1b and 2a, it can be found that the colors of all of the phases or structures in the SEM image are just opposite for those in the OM image except that the layer structures are always in grey color. The formation of this kind of layered ring-like structure can be explained by the solidification process during thixoforming. Based on the solidification of permanent mould casting ZA27 alloy[13], the solidification process of the thixoformed ZA27 alloy can be described as following. The secondarily primary phase preferentially grows on the existed primary particle surfaces without nucleation and form a shell layer on each primary particle because these two phases belong to a same phase of α'–Al. Then new nuclei will form in the liquid far away from the primary particles as the supercooling is up to the heterogeneous nucleation requirement, resulting in the formation of the individual small secondarily primary grains. Subsequently, a peritectic reaction of L+α'→β occurs and most of the secondarily primary phases including the layer structures and the individual small grains, and even part of the primary particles close to the secondarily primary phase layers become into the Zn-rich β phase. Finally, the residual liquid with eutectic compositions solidifies through the reaction L→β+η+ε. The eutectic β phase preferentially directly grows on the surface of the peritectic β phase. So the neighboring primary particles with short distance are connected together by the peritectic and eutectic β phases and only the eutectic η phase (including the ε phase) is left between the β phases in local zones, forming a kind of devoiced eutectic structures (marked by B in Figure 2a). But in the large-area regions among the primary particles, a kind of eutectic structures intersected by β and η phases (marked by A) will form due to the long atom diffusion distance. The η phase is a Zn-rich hcp phase. The solidification of the liquid pools within the primary particles is similar to that of the liquid between the primary particles. Therefore, it can be concluded that the
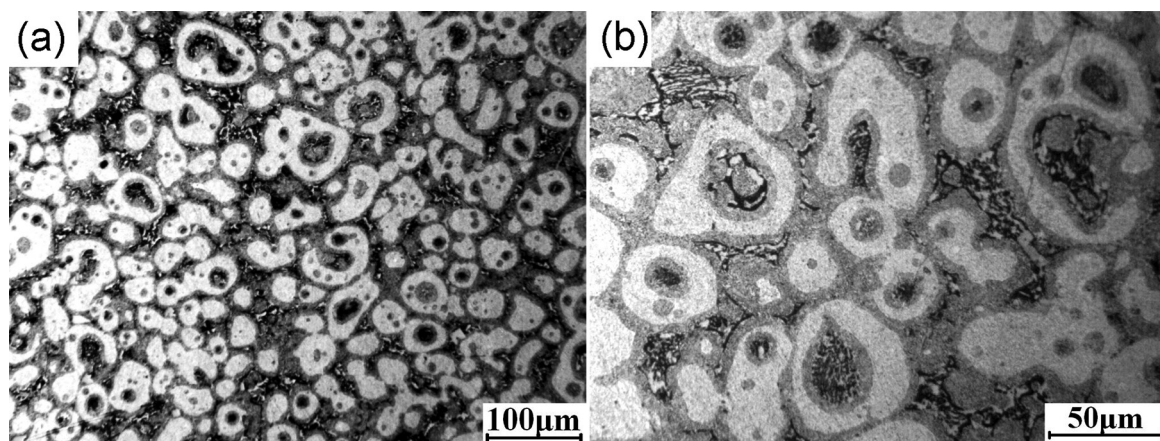

Figure 1. OM micrographs of the thixoformed ZA27 alloy. (b) is a higher-magnification image of (a).
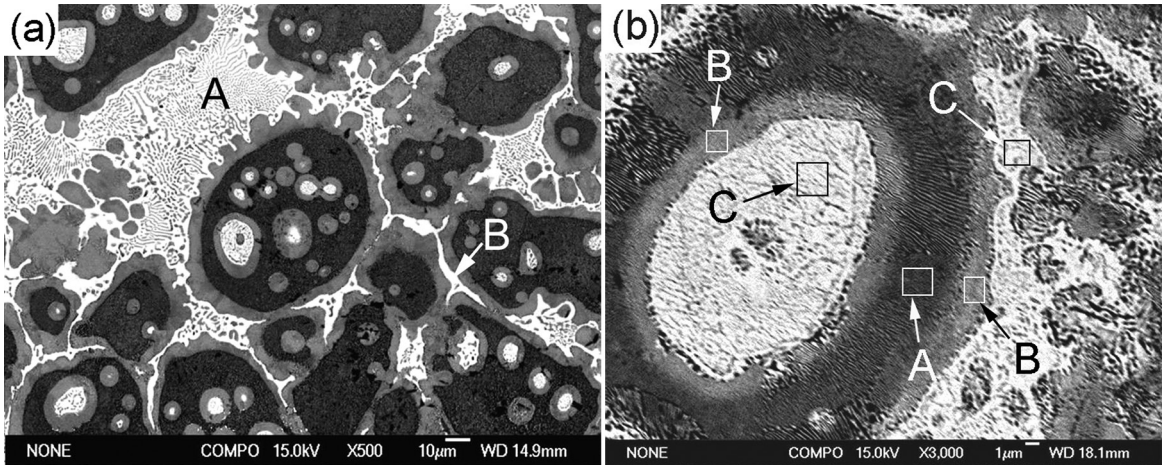
Figure 2. SEM micrographs of the thixoformed ZA27 alloy. (b) is a higher-magnification image of (a). A marked in (a) refers to regular eutectic structure and B refers to devoiced eutectic η phase. A marked in (b) refers to original α' phase, B refers to original β phase and C refers to eutectic structures.

black structures shown in Figure 2 should be the existing solid primary phase, the grey structures are the β phase and the white structures are the eutectic structures or devoiced eutectic η phase. The EDS results indicate that the black structures (marked by A in Figure 2b, a higher magnification image of Figure 2a) are rich in Al, the grey structures (marked by B) are rich in Zn and the white structures (marked by C) have a higher content of Zn (Table 1). This further demonstrates the solidification process discussed above. So it can be concluded that the general solidification process of the thixoformed ZA27 alloy is similar to that of permanent mould casting alloy although the resulting microstructures are obviously different[18].

Figure 3 shows the Al-Zn binary equilibrium phase diagram[19], which indicates that for the Zn-27Al alloy, the α' and β phases are all unstable and will decompose into the stable phases of α and η. Figure 2b shows that both the α' and β phases are not uniform single phase and they are composed of two or more phases. The XRD result also states that this alloy includes two phases of α and η besides the ε phase (Figure 4). But for the as-thixoformed alloy, large amounts of α' and β phases are still existed. All of these imply that both the α' and β phases have decomposed into the α and η phases for the presented ZA27 alloy. This characteristic is similar to that of the permanent mould casting ZA27 alloy after being naturally aged for the same time[13]. The decomposition of the α' and β phases can be expressed by the eutectoid reaction of α' (β) → α + η[10-12]. It should be noted that the Zn-rich eutectic phase in the as-thixoformed alloy is a supersaturated η phase, expressed by η_s, but that in the aged alloy is the equilibrium η phase.

That is to say that the general solidification process and subsequent decomposition characteristics of the thixoformed ZA27 alloy are basically similar to those of traditional casting alloy although the microstructures of the two-technology formed alloys are very different. Generally, the microstructure of the thixoformed alloy is composed of three structures or phases of eutectic structures (including β, η and ε phases), β phase and α' phase when the alloy has
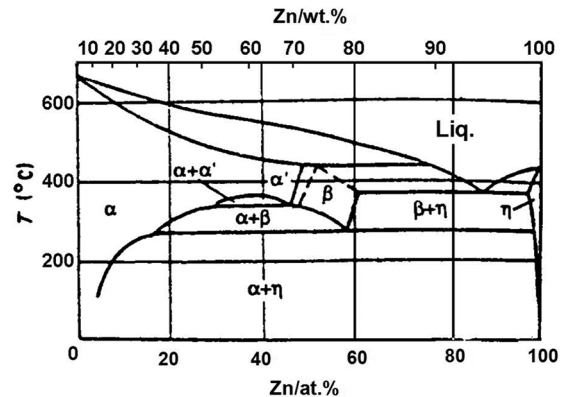

Figure 3. Al-Zn binary phase diagram[19].

Table 1. EDS results of the different positions in the microstructure.

Position	Element concentration			
	Zn	**Al**	**Cu**	**Mg**
A	48.72	48.89	1.84	0.55
B	77.06	20.08	2.07	0.80
C	95.17	3.60	0.77	0.46

just completed its solidification. But the β and α' phases are unstable and have decomposed into the α and η phases after being aged for 5 years. To verify their decomposition processes and resulting microstructure, the eutectic structures and decomposed structures of the β phase α' phases have been carefully observed in the following text.

3.2. Eutectic structures

As discussed in the above section, devoiced eutectic structures can form in thin or small-area regions between the primary particles, but a kind of eutectic structures intersected by the α and η phases will generate in large-area regions. Figure 5 gives the microstructures of two thixoformed ZA27

alloys prepared under different processing parameters[9]. It shows that most of the eutectic structures are in laminar form (marked by A) and the others are in dual-phase structures with complex morphologies (marked by B) in the large-area regions. Those in thin regions are in a devoiced form and only the eutectic η phase exists between the β phases (marked by arrows C). But for the aged alloy, the eutectic structures, especially those in the large-area regions have obviously changed in morphology. As shown by A and B in Figure 6a, the laminar-structure eutectics only exist

Figure 4. X-ray diffractograms of the thixoformed and aged ZA27 alloys.

in some small local zones (shown by arrow in Figure 6b) and most of the eutectics are in the form that lots of small black dot-like particles uniformly distribute in the white η phase matrix (Figure 6b, c). Together with the XRD result (Figure 4), it can be expected that the small dot-like black particles should be α phase. That is the eutectic β phase lamellae have decomposed into the α and η phases. It is suggested that the eutectoid η phase preferentially attaches on the neighboring eutectic η lamellae without renucleation during decomposition of the thin β lamellae. To decrease the α/η interface energy, the eutectoid α phase will become into the small particles. Namely, the original laminar eutectics have transformed into the structures that small α particles uniformly distribute in the η matrix. But in the thin regions as shown by arrow C in Figure 6a, the devoiced η phase structures almost do not change (Figure 6d).

The two phases of the eutectic structures, β and η, all belong to metallic phase and their volumes in the eutectic structures are comparative as shown by Figure 5. So they preferentially grow up in regular laminar form during solidification. Although the β phase (including the peritectic β phase) tends to decompose into α+η laminar structures (this will be discussed in the following section). But it can be expected that the original β phase lamellae are very thin and their decomposed α+η lamellae are thinner. The interface energy of such microstructure should be very large. In contrast, the interface energy can be more rapidly decreased if the thin eutectoid α lamellae change into spheroidal particles besides that the eutectoid η phases directly grow up from the neighboring eutectic η lamellae during the long-time ageing.
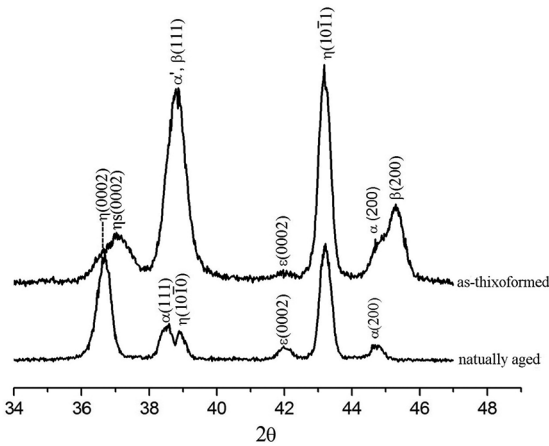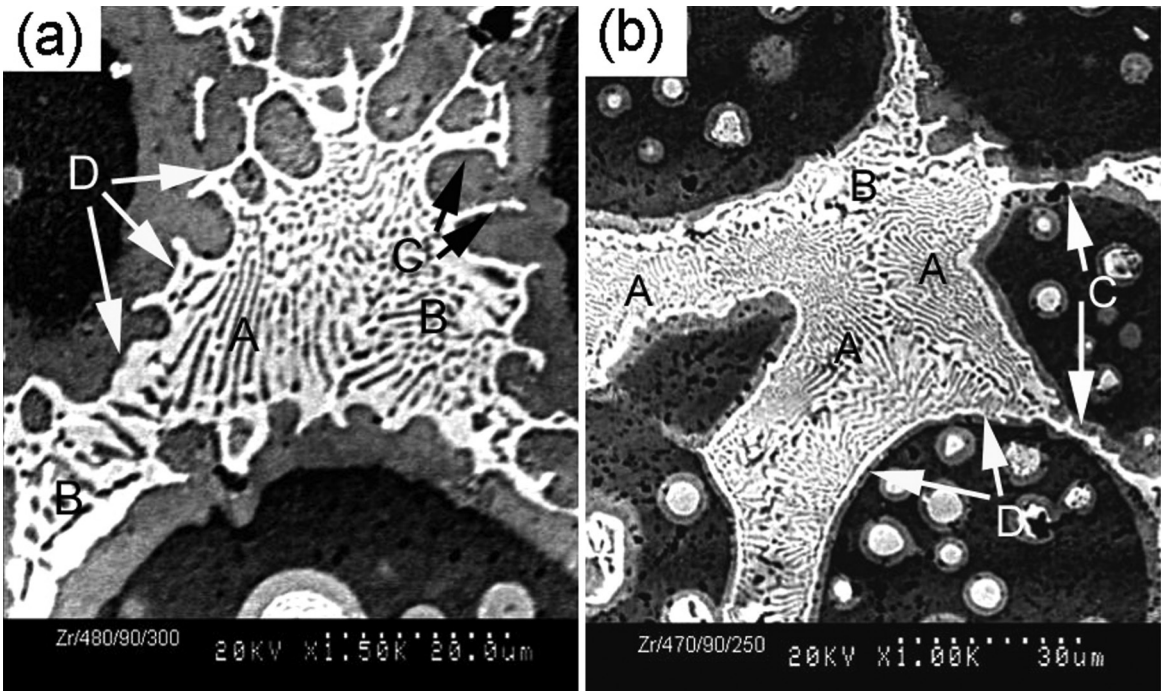

Figure 5. SEM micrographs of the as-thixoformed ZA27 alloys under different processing parameters[9]. (a) reheating temperature: 480 °C, reheating time: 90 min, mould temperature: 300 °C; (b) reheating temperature: 470 °C, reheating time: 90 min, mould temperature: 250 °C. A refers to regular lamellar eutectic structures, B refers to irregular eutectic structures, C refers to devoiced eutectic η phase and D refers to eutectic η phase layer.
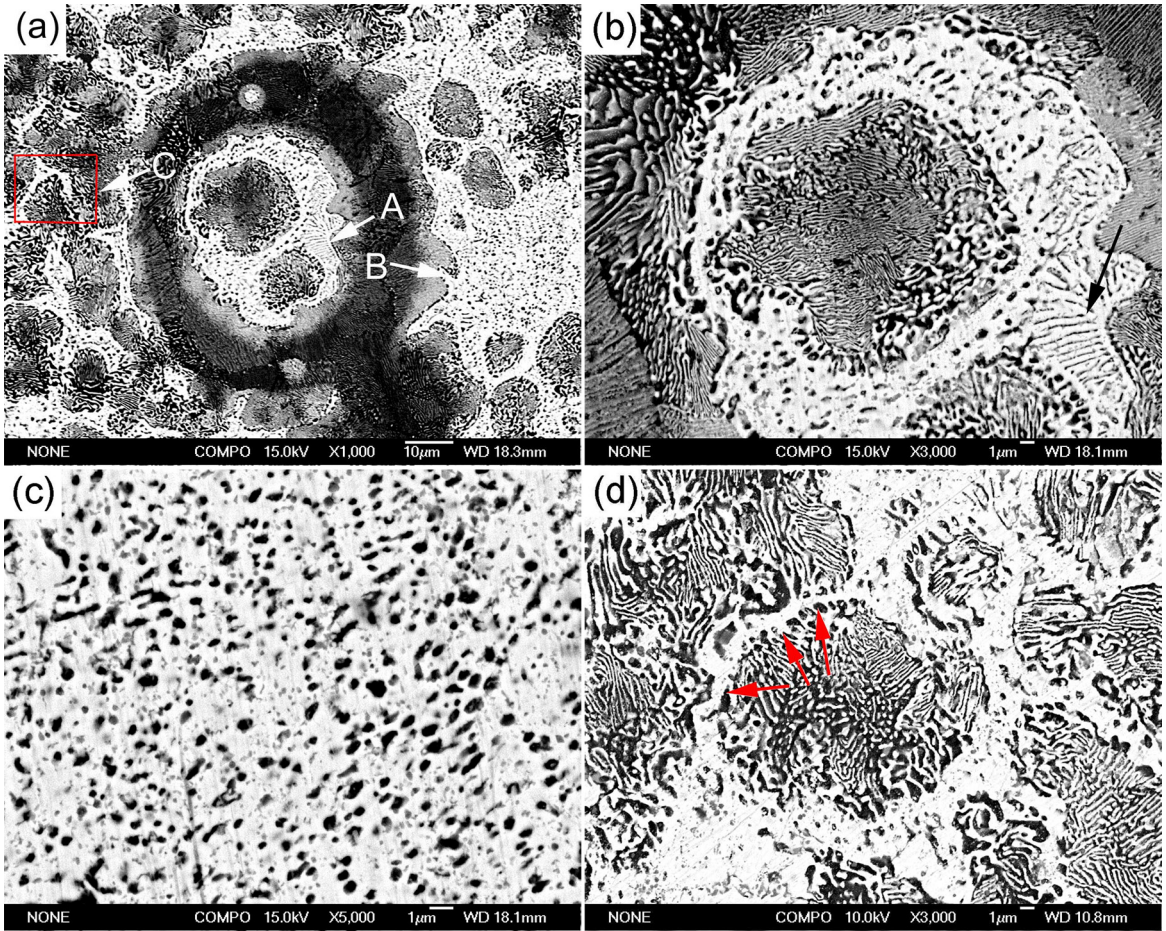
Figure 6. SEM micrographs of the as-thixoformed ZA27 alloys. (b-d) are the higher-magnification images of different regions of (a) and show the different morphologies of eutectic structures. A and B refer to regular laminar eutectic structures, and C refers to devoiced η phase.

That is, the original β + η laminar eutectics have changed into a structure that small α particles uniformly distribute in η phase matrix and the devoiced η phase structures almost have not changed after being naturally aged for 5 years.

3.3. Decomposition microstructure of β phase

Figure 7a shows that both the α' and β phases all have decomposed into the regular two-phase structures and the local high-magnification image in Figure 7b indicates that this kind of regular two-phase structure is a laminar structure like regular laminar eutectics. Together with the above XRD result, it can be suggested that the white lamellae are η phase and the black lamellae are α phase. From the above Section 3.1, it is known that the β phase is close to the eutectic structures. Figure 7c, d show that there is a quite continuous black α layer or a line of particle-like α layer between the eutectics and the β phase decomposition products.

For the as-thixoformed alloy, there is always a continuous η phase layer on the β phase surface (marked by arrows D in Figure 5). During solidification, the liquid phase close to the β phase should be rich in Zn and a Zn-rich η phase layer will form on the β phase surface during eutectic reaction so that Zn content in the liquid can be decreased rapidly. The continuous η phase layer has not likely changed as

shown by the red dot-lines in Figure 7c, d although the laminar eutectic structures close to it have obviously altered. According to their location, the continuous and dot-line α phases are originated from the β phase. The β phase close to the Zn-rich η phase layer should have high content of Al and is an Al-supersaturated solid solution, and the closer to the η phase layer, the higher the Al solubility. So Al-rich α phase will first precipitate in the β phase close to the eutectic η phase layer during natural ageing, forming the continuous or dot-line α phase. The formation of the continuous or dot-line α phase should be related to the Al content in the β phase. The higher the Al content, the more continuous the α phase. The precipitation of the α phase must increase the Zn content in the β phase and promote the nucleation and subsequent growth of eutectoid η phase (marked by arrows A in Figure 7c). In addition, the eutectoid η phase also preferentially grows up from the eutectic η phase surface without renucleation (marked by arrows B). The formation and growth of the η phase then accelerate the nucleation or direct growth of eutectoid α phase from the surface of the continuous or dot-line α phase. In this way, two-phase α + η of the eutectoid reaction then form and grow towards the β phase inside through their mutual coordination and acceleration, forming the regular laminar structures. Figure 7d
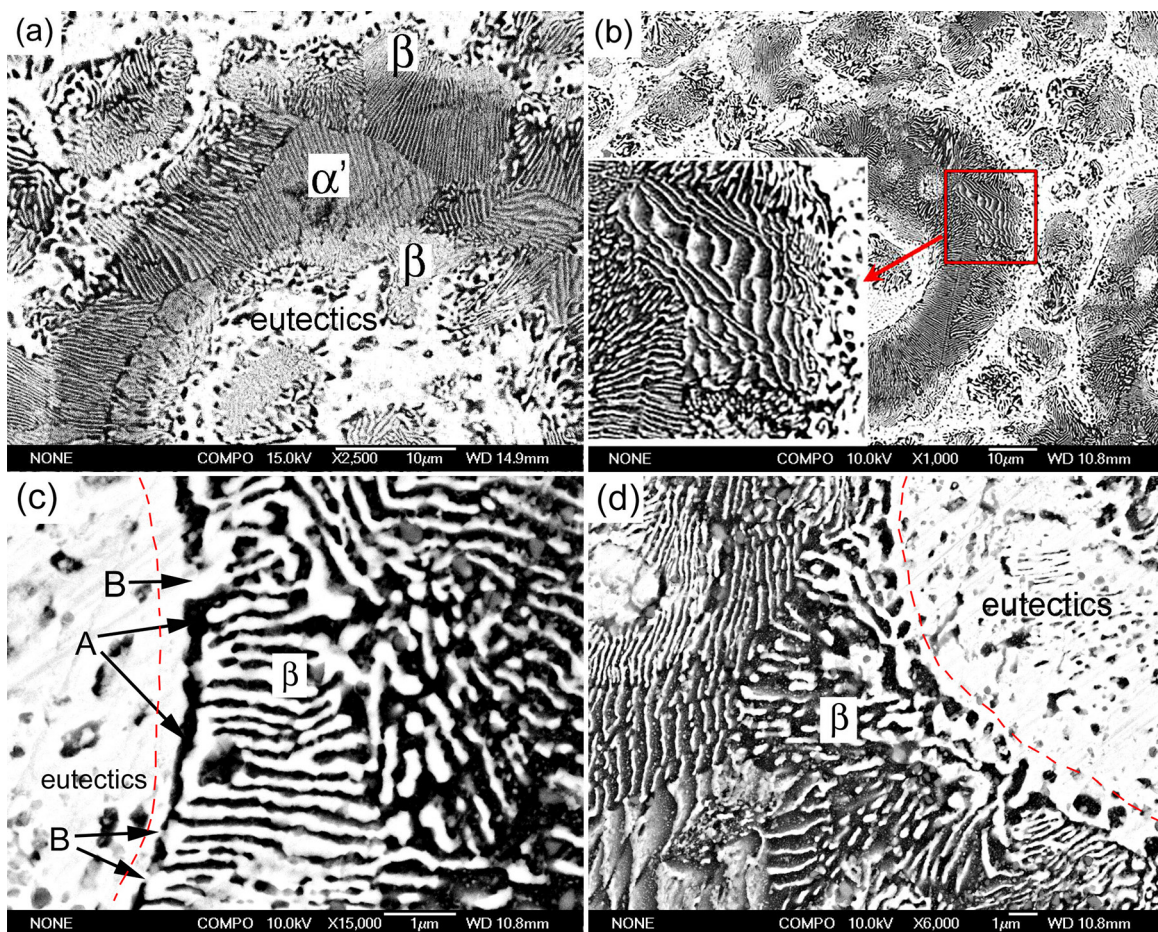
Figure 7. SEM micrographs of the thixoformed ZA27 alloy. (a) shows the general morphologies of both the original α' and β phases, the local high-magnification image in (b) shows the laminar structure characteristic of the β phase decomposition products, and (c) and (d) show the continuous black α layer and the line of particle-like α layer between the eutectics and the β phase decomposition products respectively. A marked in (c) refers to renucleated and subsequently grown η phase and B refers to directly grown η phase from the existing eutectic η phase surface.

shows that the morphology of the formed α + η eutectoids is not always in the regular laminar form, but in a quite complicated shape in some local zones. The white η phases appear in small broken lamellae. Similar to the change of the regular η + β eutectics discussed in Section 3.2, the variation of the eutectoid structures in this way should also be attributed to decrease the α/η interfacial energy. So it can be supposed that the laminar eutectoid structures are unstable and the η phase has a tendency of changing from laminar shape to particle form.

In summary, the β phase is unstable and will decompose into regular α + η laminar structures through eutectoid reaction during natural ageing. A continuous or dot-line α phase first precipitates in the β phase close to the eutectic η phase layer. Then eutectoid η phases nucleate or directly grow up on the eutectic η phase layer, which promote the nucleation or direct growth of eutectoid α phase from the existing continuous or dot-line α phase. Subsequently, the resulting α + η phases grows towards the β phase inside, forming the regular laminar α + η structures. But the η phase has a tendency of changing from laminar shape to

particle form, which leads the eutectoids to become into the structures with complicated morphology as the ageing further proceeds.

3.4. Decomposition microstructure of α' phase

Figure 8a shows that both the marginal β phase and the center α' phase all have decomposed into regular α + η laminar structures. In view of the laminar structures, there is no difference between the products decomposed from these two phases and the center α' phase' products are the continuation of the marginal β phase' products (Figure 8b). That is, the α' phase' products are originated from the continuous growth of the β phase' products towards the center α' phase and they do not form from renucleation. The largest difference is that the color of the α' phase' products is darker than that of the β phase' products (Figure 8a, b) because the Al content in the original α' phase is higher than that in the original β phase (Table 1). In addition, the lamellae from the former phase are coarser and more irregular than those from the latter phase.
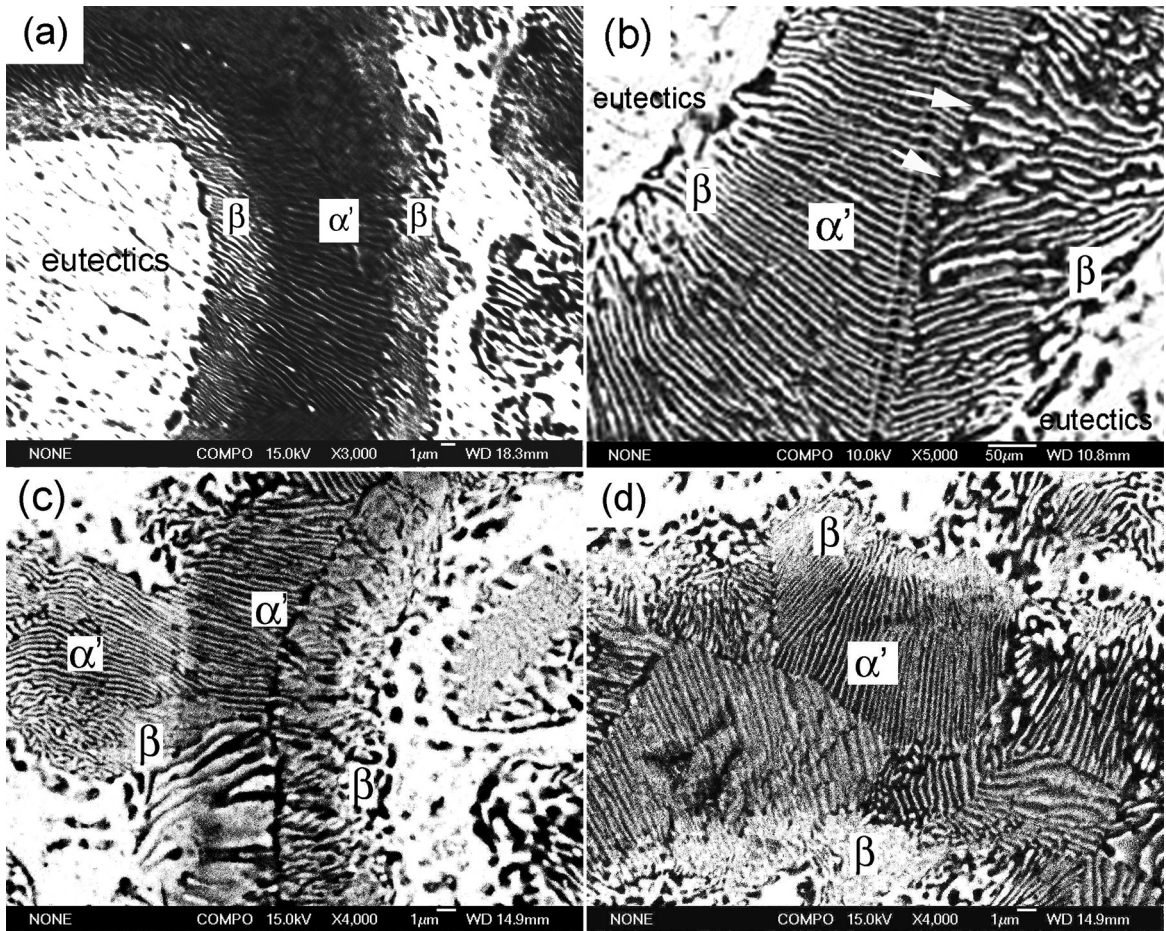
Figure 8. SEM micrographs of the thixoformed ZA27 alloy. (a) and (b) show that the α' phase' products are originated from the continuous growth of the β phase' products towards the center α' phase, (c) and (d) show that an obvious boundary can form when two eutectoid cells grow to impinge.

In fact, the β phase close to the α' phase is transformed from the α' phase through the peritectic reaction of L+α'→β and this two phases all belong to fcc structure[10]. Only the compositions of the β and α' phases, especially their Al contents are different. These provide the basis for the α' phase products to form from the continuation of the side β phase' products without renucleation. Previous investigations indicated that the β phase was more unstable than the α' phase and the β phase would decompose in a short time[11]. The present results also shows that the decomposition of the β phase is earlier than that of the α' phase. As discussed in Section of 3.3, the laminar structures from the β phase are unstable and the η lamellae have a tendency of changing into particle-like structures. In fact, the essential of this change is a coarsening process driving by deceasing interface energy. It is just due to this change that leads the laminar structures from the β phase to be coarser and more irregular than those from the α' phase.

As shown by Figure 8b, an obvious boundary has formed when the two-side products grow to impinge (marked by arrows). This phenomenon can be more clearly seen in Figure 8c. But Figure 8d indicates that it is not true that there

is only one boundary in the primary particles, there can be several ones. It can be expected that for a hollow spheroidal primary particles, the eutectoid cells can form on the whole inner and out surfaces and grow towards the center α' phase regions. Several boundaries will form when they impinge each other due to their different growth directions.

Besides the eutectoid structures with irregular laminar form, Figure 9 shows that there are other-morphology eutectoids, such as the structure with small η phase particles uniformly distributed in the α matrix (Figure 9a) and the structure with broken small η phase lamellae or rod-like η phases (marked by the red rectangle in Figure 9b). The reason that generates these structures, similar to that resulting in the non-laminar eutectics and irregular β phase' products discussed above, should be also attributed to the coarsening and spheroidization of the η phase lamellae.

Figure 10 shows that there are some boundaries in the α' phase' products (marked by arrows). But it indicates that these boundaries are very different from those shown in Figure 8b-d. The laminar morphologies and laminar distances in the two sides of the boundaries shown in Figure 8b-d are obviously different. As discussed above, these boundaries are formed
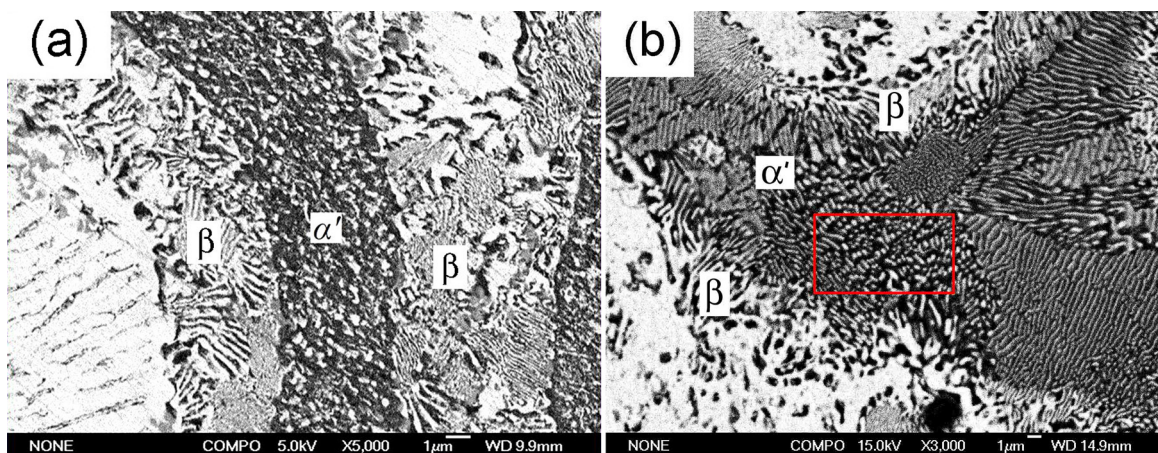
Figure 9. SEM micrographs of the thixoformed ZA27 alloy. Showing the non-laminar morphologies of the α' phase decomposition products, (a) the structure with small η phase particles uniformly distributed in the α matrix and (b) the structure with broken small η phase lamellae or rod-like η phases.
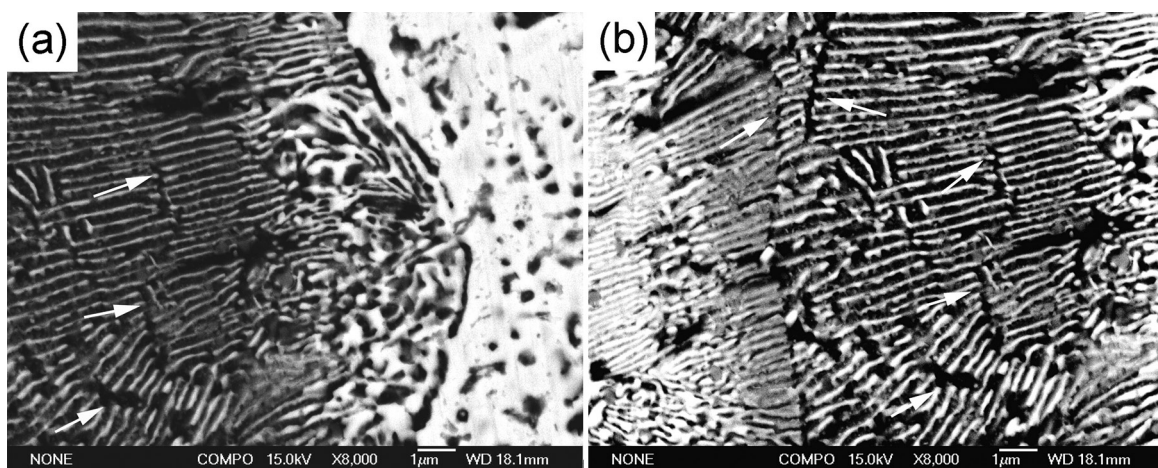

Figure 10. SEM micrographs of the thixoformed ZA27 alloy. Showing crack characteristics within the decomposition laminar products from α' phase as shown by arrows.

from impingement of different eutectoid cells. But those shown by Figure 10 are almost same and the boundaries seem to be formed from breaking of the laminar structures. It can be suggested that the volumes of the structures before and after decomposition are possible different, which can generate stress in the decomposed structures during ageing. Cracks will form when the stress exceeds the local ultimate strength. Durman et al. also suggested that internal stress may generate during eutectoid reaction for permanent mould casting ZA27 alloy[10]. But if the resulting stress concentration can generate cracks and the "boundaries" are cracks should be further verified.

Therefore, it can be concluded that the decomposition products of the α' phase also appears in regular α + η laminar shape and they form from the continuation of the β phase towards the center α' phase. Boundaries will form when the laminar cells with different orientations grow to impinge. The laminar structures are also unstable and the η phase lamellae will coarsen and spheroidize as the ageing time increases. In addition, the laminar structures may crack due to internal stress.

4. Conclusions

(1) The β and α' phases of the thixoformed ZA27 alloy are unstable and have decomposed after being naturally aged for 5 years. The general decomposition characteristics are similar to those of permanent mould casting alloy.

(2) Most of the original β + η laminar eutectics have change into a structure that small α particles uniformly distribute in η phase matrix and the devoiced η phase structures almost have not changed.

(3) During the natural ageing, a continuous or dot-line α phase first precipitates in the β phase close to the eutectic η phase layer. Then the eutectoid η phases nucleate or directly grow up from the eutectic η phase layer, which promotes the nucleation or the direct growth of the α phases from the existing continuous or dot-line α phase. Subsequently, the resulting α + η phases grows towards the β phase inside, forming the regular laminar α + η structures.

(4) The decomposition products of the α' phase also appears in regular α + η laminar shape and they form from the continuation of the β phase towards the center α' phase. Boundaries will form when the laminar cells with different orientations grow to impinge. In addition, the laminar structures may crack due to internal stress.

(5) The eutectoid η phase lamellae have a tendency of changing from laminar shape to particle form, which leads the eutectoids to become into non-laminar structures.

Acknowledgements

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