CHINA FOUNDRY Research & Development
Vol.13 No.1 January 2016
DOI: 10.1007/s41230-016-5109-8

Compressive characteristics of closed-cell aluminum foams with different percentages of Er element

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Abstract: In the present study, closed-cell aluminum foams with different percentages of erbium (Er) element were successfully prepared. The distribution and existence form of erbium (Er) element and its effect on the compressive properties of the foams were investigated. Results show that Er uniformly distributes in the cell walls in the forms of Al_3Er intermetallic compound and Al-Er solid solutions. Compared with commercially pure aluminum foam, Er-containing foams possess higher micro-hardness, compressive strength and energy absorption capacity due to solid solution strengthening and second phase strengthening effects. Additionally, the amount of Er element should be controlled in the range of 0.10wt.%–0.50wt.% in order to obtain a good combination of compressive strength and energy absorption properties.

Key words: aluminum foams; erbium element; compressive property; melt foaming method

CLC numbers: TG136˙.1 **Document code:** A **Article ID:** 1672-6421(2016)01-036-06

Metallic foams are one of the latest inventions in the field of materials. Due to their excellent combination of mechanical and physical properties, some preparation methods have been developed [1-3] and the potential and practical applications of closed-cell aluminum foams have been systematically reviewed [4]. Recently, there is a considerable increasing interest in using closed-cell aluminum foams as lightweight structural components or energy absorption parts, specifically for automobile, railway, shipbuilding and aerospace applications [5]. Nevertheless, in these application fields, commercially pure aluminum foams are seldom used, as their relatively lower mechanical properties limit their widespread applications [6-8]. Researchers have noticed this problem and some investigations have been carried out. Huang et al. studied the effect of Sc element on the quasi-static compressive properties of aluminum foam and the results showed that Sc element can dramatically improve yield strength of aluminum foam owing to the effect of Al_3Sc precipitates [9]. In our previous research, Mn elements were added to aluminum foams and much higher compressive properties were obtained [10]. The well-known high cost of the Al-Sc alloy will limit the economical efficiency of the products. Compared with Sc element, Mn element is much cheaper. While it is important to develop different types of economical products from the engineering application viewpoint, we therefore aim at to seek out other economic ways to produce high strength closed-cell aluminum foams. Generally, it is accepted that the mechanical properties of aluminum foams mainly rely on the properties of cell wall materials and the relative density and microstructure of the foams [11-14]. As an ideal addition, Er element (which is much cheaper than Sc element) has been widely used in aluminum alloys and it has been confirmed that Er element can improve the mechanical strength of Al-Zn-Mg and Al-Mg alloys [15-18]. It is noted that most available studies focus on bulk metals and few studies have focused on the effect of Er element on the mechanical properties of porous materials, which possess different kinds of solidification processes and heat transfers compared with bulk metals. Therefore, further research is needed. In the present study, Er element in the form of Al-30wt.% Er master alloy was introduced into aluminum foams and the effect of Er element on the compressive characteristics of the foams was investigated.

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Received: 2015-10-30; Accepted: 2015-11-30

1 Experimental procedures

1.1 Specimen preparation

The matrix material used was commercially pure aluminum ingots (with purity of ~99.5%). In addition, Ca granules (commercially pure, granularity between 1–2.5mm), TiH_2 (commercial pure, 300±20 mesh) and Al-30wt.% Er master alloy (commercially available) were used as thickening agent, foaming agent and addition agent, respectively. Melt foaming method was applied to fabricate the foams and the process is briefly described in the following steps: (1) melting certain quantity (~1 kg) of aluminum ingots in a low carbon steel crucible to a fixed temperature; (2) adding different percentages of Er element and certain quantity of Ca granules (2.wt.%) to the melt accompanied by stirring to make Er element and thickening agent homogeneously distribute in the melt; (3) adding certain amount of TiH_2 (0.80%) to the melt accompanied by stirring to make the foaming agent distribute in the slurry uniformly; (4) holding the melt for some time and then air cooling the flux after it was foamed. During the whole process except for the last step, the melt temperature was maintained at 680±2 °C.

1.2 Parameters calculation method

Specimens for compression testing were machined to the size of 20 × 20 × 20 mm^3 by electro-discharging machine. Meanwhile, density and porosity of all the specimens were calculated using Equation (1) and Equation (2):

$$\rho = \frac{m}{v} \quad (1)$$

$$p = \frac{(\rho_0 - \rho)}{\rho_0} \quad (2)$$

where v is the volume of specimen (mm^3), m is the weight of specimen (g), ρ is the density ($g \cdot mm^{-3}$) of specimen, ρ_0 is the density of the matrix material and p is porosity of specimen. Analytical balance (with the precision of 0.0001 g) and digital caliper were used for weight measurement and accurate dimension measurement, respectively. Mean intercept length technique was applied to determine the mean pore size of the foams.

1.3 Morphology observation

Typical metallographic sample preparation method was used for microstructure morphology observation. Specimens were ground with 400, 600, 800, 1500 and 2000 grit emery paper, polished using 0.25 micron diamond paste, and washed with ethyl alcohol, then washed with ethyl alcohol again after etching with Keller reagent and finally dried with cold flowing air. The microstructures and the distribution of Er element were observed using a Hitachi S4800 scanning electron microscope (SEM) equipped with energy dispersive X-ray spectrometer (EDS) and a Phenom G2 Pro desktop scanning microscopy (SEM). Phase constituents were analyzed using XRD (SmartLab, Rigaku) with CuKa radiation.

1.4 Mechanical test

Uniaxial quasi-static compression tests were carried out using a SUNS Electron Universal Material Testing Machine (SHT5305), with a maximum load of 300 kN. Here, testing with an initial strain rate of 0.001 per second was performed at room temperature. Vaseline was used to minimize the friction between the specimen and the plates. Displacement control was used to conduct the compression tests. Load and displacement values obtained by sensors were recorded automatically by a computer. Engineering stress σ was defined as the load (kN) divided by a specimen original cross-section area; engineering strain ε was defined as the displacement (mm) divided by specimen original height. SHIMADZU micro-hardness tester, with a load of 10 g for 15 s, was applied to obtain the variation tendency of cell wall micro-hardness. The average of at least 15 randomly selected points was considered for cell wall micro hardness measurement.

2 Results

2.1 Characteristics of foams

Figure 1(a) shows a representative sample of the foams with Er percentage of 0.30wt.%. It is clear that the melt with Er element possesses good foaming behavior. Figure 1(b) shows a typical cross section image of closed-cell aluminum foam with Er percentage of 0.30wt.%. It is evident that the cell size in the foam varies in a narrow range and it appears to be quite uniform except for one or two relatively larger pores. Meanwhile, the pores are spherical, separated and closed-cell. The pore size and porosity of the foam prepared using the method described

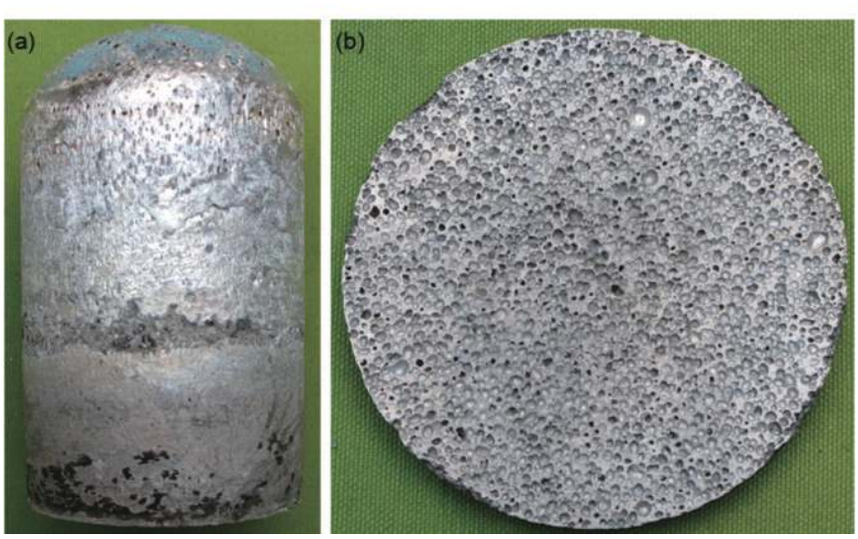

Fig. 1: Sample (a) and cross-section (b) images of closed-cell aluminium foam with 0.30wt.% Er

above mainly distribute between 1.5–2.5 mm and 72%–80%, respectively.

2.2 Micro-hardness variation

Figure 2 shows the micro-hardness (Vickers hardness) variation tendency of the cell walls with different percentages of Er element. The average micro-hardness values for the foams with Er percentages of 0.00, 0.05, 0.10, 0.30, 0.50 and 0.90wt.% are about 35, 40, 43, 49, 45 and 40, respectively. On the whole, Er element enhances the cell wall micro-hardness. Meanwhile, it is clear that the cell wall micro-hardness gradually increases at first and then decreases moderately with the increase of Er percentage.

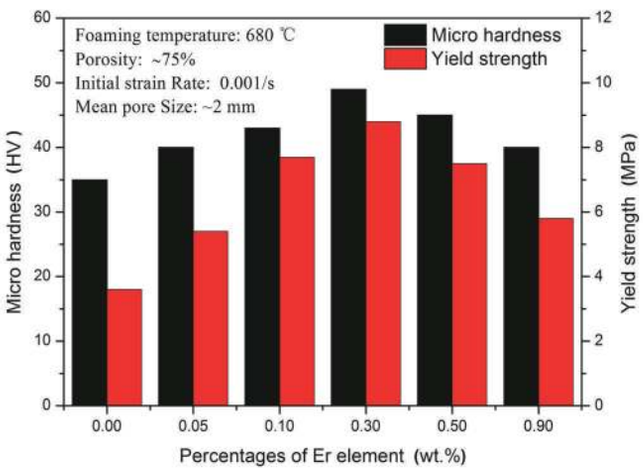

Fig. 2: Variation tendency of yield strength and micro-hardness of foams with different Er contents

2.3 Compressive and energy absorption properties

Quasi-static compressive engineering stress (σ)-engineering strain (ε) curves of the specimens, with uniform porosity (~75%) and mean pore sizes (~2 mm), with different percentages of Er element, are shown in Fig. 3(a). In all cases, the stress (σ)-strain (ε) curves exhibit three universal deformation stages: linear deformation stage (I) where the stress increases linearly with the increase of strain due to the elastic bending of cell wall; then a plateau deformation stage (II) where the stress remains constant or increases slightly with the strain increasing due to the strain hardening effect; finally a densification stage (III) emerges where the stress increases steeply along with the strain increasing due to the collapsed cells being almost fully compacted together. It is obvious that Er element has an important effect on the compressive properties of foams. Figure 2 also shows the variation tendency of the yield strength for the foams. As is known, yield strength (defined as the first peak stress on stress-strain curve) is an important aspect to estimate the mechanical properties of metallic foams. The values for the foams with Er percentages of 0.00, 0.05, 0.10, 0.30, 0.50 and 0.90wt.% are about 3.6, 5.4, 7.7, 8.8, 7.5 and 5.8 MPa, respectively. This means that Er element can significantly improve the yield strength of foams especially in foams with Er contents of 0.10, 0.30 and 0.50wt.%, where the values are more than two times that of the foams without Er element. It should also be noted that with excessive Er element, the yield strength decreased gradually. In addition, the stress-strain curves can be divided into four categories, i.e. the commercially pure foam (without Er), foams with Er percentage of 0.30wt.%, foams with Er contents of 0.05wt.% and 0.90wt.%, and foams with Er contents of 0.10 wt.% and 0.50wt.%. The variation tendencies of the curves for the latter two categories are almost the same (the curves are average data of three individual specimens). Meanwhile, the mean plateau strength (defined as the mean stress between the strain of 0.1-0.5) of the foams first increases and then decreases with the increase of Er percentage. It can be seen that the mean plateau strength of the third category (foam with 0.05wt.% or 0.9wt.% Er) is almost two times that of the first category (foam without Er element), which is equivalent to some of the aluminum alloy foams [19, 20]. The values of the second (foam with 0.30% Er element) and fourth categories (foams with 0.10wt.% or 0.50wt.% Er) are basically the same and reach a maximum under the experiment condition. Further,

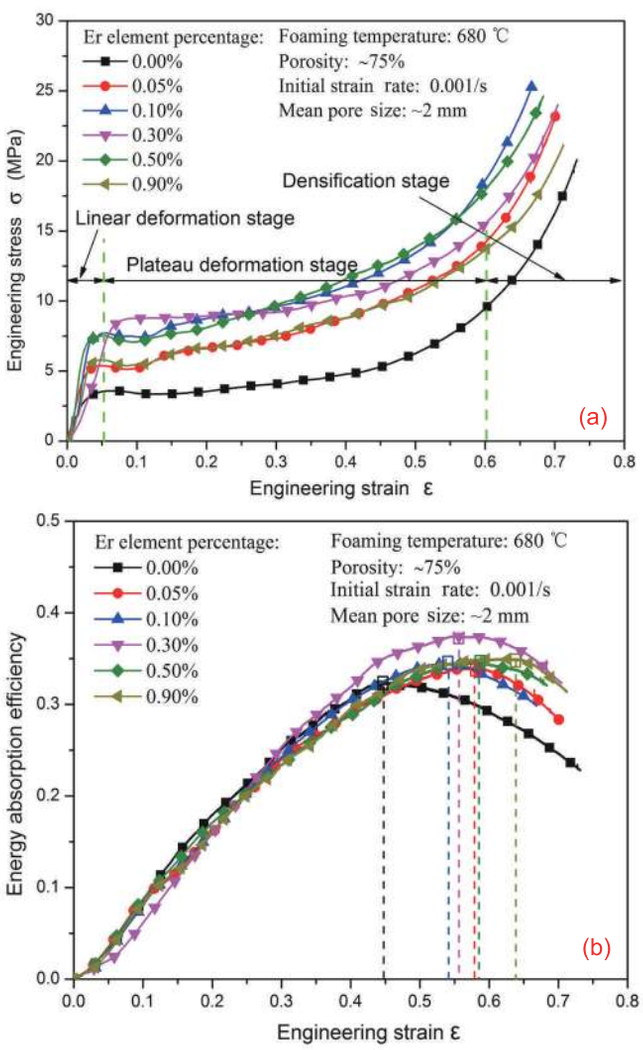

Fig. 3: Engineering stress-strain curves for foams with different percentages of Er element against deformation stages (a) and energy absorption efficiency (b)

Er element affects the densification strain of the foams to some extent. The densification strain is usually defined as the strain corresponding to the maximum of the energy absorption efficiency [21]. The energy absorption efficiency is calculated using Equation (3) [22] and Equation (4) [23]:

$$W = \int_0^{\varepsilon} \sigma d\varepsilon \quad (3)$$

$$I = \frac{W}{\sigma} \quad (4)$$

where I is the energy absorption efficiency, W is the energy absorption capacity of specimen, σ is the stress and ε is the strain. Figure 3(b) shows the energy absorption efficiency curves of foams with different Er contents. It is obvious that the values of densification strains corresponding to the maximum of the energy absorption efficiency can be obtained according to the definition of densification strain. The densification strains of the curves for the foams with Er percentages of 0.00, 0.05, 0.10, 0.30, 0.50 and 0.90wt.% are about 0.45, 0.58, 0.54, 0.56, 0.59 and 0.64, respectively. It is thus concluded that adding Er element can increase the densification strain of the foams to some extent compared with pure foams without Er element under the present conditions.

In most cases, the energy absorption property of metal foams plays an important role in industrial applications. Moreover, energy absorption capacity is one of the most important aspects in evaluating the property. It can be seen from Fig. 4 that in all cases, energy absorption capacity (calculated using Equation 3) grows with the increase of strain just as other metal foams [13, 22]. In the initial stage, there is no obvious difference among the six groups of specimens, but the amounts of energy absorbed in the middle stage are significantly different. Similar with the effect on the engineering stress-engineering strain (as shown in Fig. 3a), energy absorption capacity can be divided into three levels: commercially pure aluminum foam, foams with Er contents of 0.05wt.% and 0.90wt.%, foams with Er contents of 0.10wt.%, 0.30wt.% and 0.50wt.%. The energy absorption capacity for the

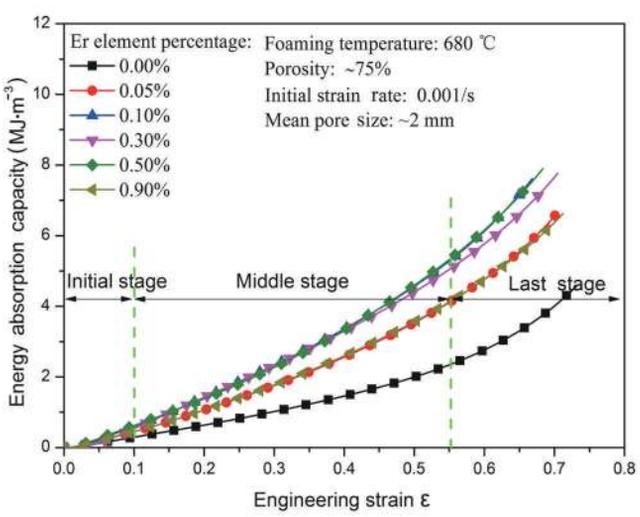

Fig. 4: Energy absorption capacity of foams with different percentages of Er element

foam without Er element is about 1.70 MJ·m^{-3}, where the strain is 0.45; while the values for the foams with Er contents of 0.05, 0.10, 0.30, 0.50 and 0.90wt.% are about 3.02, 3.93, 3.83, 3.93 and 3.05 MJ·m^{-3}, respectively, where the strain is about 0.45. All these results show that a small amount of Er element can greatly improve the energy absorption capacity of commercially pure foams (without Er element). Also, it should be noted that the energy absorption capacity increased first and then decreased with the increase in percentages of Er element. Under present conditions, specimens with Er contents of 0.10wt.%, 0.30wt.% and 0.50wt.% possess a good combination of plateau strength and energy absorption capacities. In addition, specimens with Er content of 0.30wt.% possess an optimal yield strength (as shown in Fig. 2). From the above discussion, energy absorption capacity of foams is closely related to the variation of stress within a certain strain range according to equation (3). The value is equal to the area surrounded by the stress-strain curve, the horizontal axis and a straight line perpendicular to a strain. As shown in Figs. 3(a) and 4, energy absorption capacities of aluminum foams with Er content of 0.10wt.%, 0.30wt.%, 0.50wt.% are not much different under low strain. Under high strain, the hardening phenomenon of foams with Er content of 0.10wt.% and 0.50wt.% is more obvious compared with foam with Er content of 0.30wt.% during compression. Therefore, the values of energy absorption capacities of foams with Er content of 0.10wt.% and 0.50wt.% are higher than foams with Er content of 0.30wt.% under high strain.

3 Discussion

Figure 5 shows the SEM images (obtained by Phenom G2 Pro desktop scanning microscopy (SEM)) of as-received Al-30wt.% Er master alloy. It can be clearly seen that the microstructure consists of α-Al matrix and AlEr intermetallics. The AlEr intermetallics exist in the forms of blocks and strips and uniformly distribute in the α-Al matrix.

In order to investigate the mechanism of Er element on the mechanical properties, the existence forms of Er element in the foams should be explored. EDS detection of the grain boundaries [Fig. 6(a and b)] and surface scanning [Fig. 6(c and d)] on the cell walls (with Er percentage of 0.30wt.%) were applied and the results are shown in Fig. 6. The point spectrum analysis was also applied on other regions of the grain boundaries and the results clearly show that part of the Er element segregates on the grain boundaries in the forms of strips with shallow colour, while the regions with darker colour mainly consist of Al and Ca element. According to the Al-Er binary phase diagram, the Al-30wt.% Er master alloy has a hypereutectic composition. In addition, the surface scanning results [as shown in Figs. 6(c) and (d)] show that Er element uniformly distributes in the cell walls as well as on the grain boundaries. It is known that the solubility of Er element in Al matrix is less than 0.05wt.% at room temperature. When the melt is foamed and then air cooled, part of the Er element will exist in the form of supersaturated solid solution. Thus, when the concentration of Er element approaches

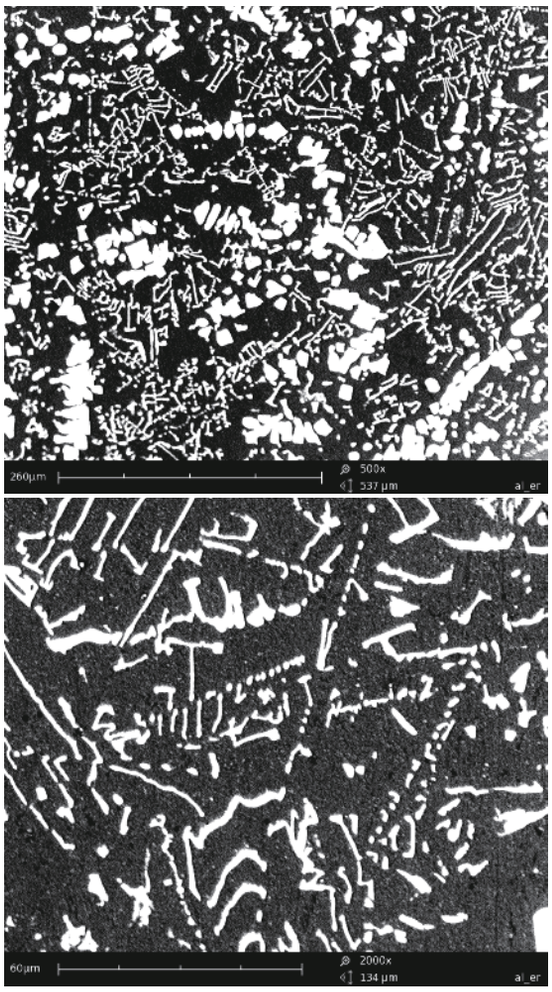

Fig. 5: SEM images of as-received Al-30wt.% Er master alloy

the eutectic point, Al_3Er compounds will form, serving as part of the eutectic structure [24].

To further confirm this process, XRD detections were used and the results are shown in Fig. 7. The main difference between Er-containing foams and commercially pure foams is the emergence of Al_3Er phase in the Er-containing foams, which corresponds well with the results described above. The results mentioned above mean that Er element exists in the forms of primary Al_3Er phase and Al-Er solid solution in the cell walls. Thus, the enhancement of micro-hardness, yield stress and plateau strength of the Er-containing Al foams can be attributed to the reasons in the following discussion.

During the preparation process, Er atoms diffused into the crystal lattice of commercially pure aluminum, resulting in the lattice distortion of aluminum atoms and the increasing of the dislocation density. It is known that the deformation of metal material mainly relies on the dislocation slip. Thus, during the compressive process, more dislocations will be evident. Additionally, Er possesses the largest lattice parameter misfit with aluminum atom, which will strongly retard the motion of the dislocations [25, 26]. Furthermore, Er element can dramatically refine the dendritic structures of aluminum and improve the micro-hardness [15]. Meanwhile, Al_3Er phase forms with a stable L12 crystal structure, which will improve the mechanical properties of aluminum alloys due to the refinement and spheroid of α-Al dendrite [26]. Additionally, the precipitated Al_3Er particles in the Al matrix can effectively enhance the micro-hardness of aluminum alloys. All these are beneficial to the deformation process of the Er-containing foams. Hence, the cell wall micro hardness, yield strength and plateau strength of the foams are intensified and then the yielding, buckling and friction of the cell walls are improved, leading to a higher energy absorption capacity. However, excessive Er content will reduce the mechanical performance of the foams and this may be due to the fact that the increasing addition of Er results in too many inclusions, some of which with large size and agglomerated distribution may act as crack sources during compressive deformation, and hinder the further improvement of the mechanical properties.

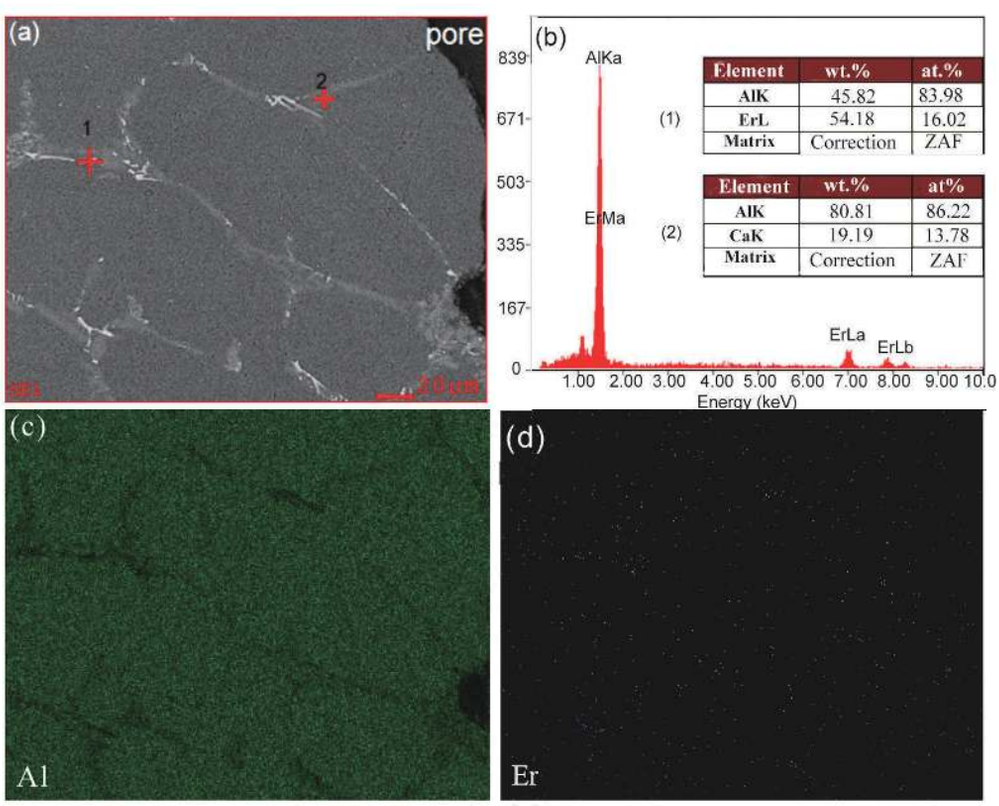

Fig. 6: EDS detection results of foam with 0.03wt.% Er element grain boundaries (a) and (b). Surface scanning of elements Al (c) and Er (d)

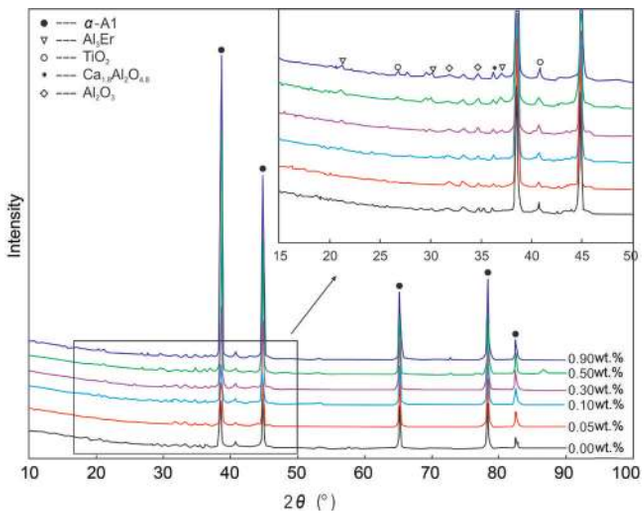

Fig. 7: Phase compositions of foams with different Er contents

The settling of some particles is another factor in the experiment results. These particles tend to settle at the bottom of crucibles, resulting in the formation of sludge and reducing the overall efficiency of the additions. Under the present condition, compressive properties and energy absorption capacities of aluminum foams with Er contents of 0.10wt.%, 0.30wt.% and 0.50wt.% are greatly improved compared with pure aluminum foams. In consideration of the cost of production and the mechanical properties, adding 0.10wt.%–0.50wt.% Er elements to commercially pure Al foams will be more appropriate.

4 Conclusions

Metal foaming method was used to produce Er-containing foams and the effect of Er element on the quasi-static compressive behaviours of aluminum foams were investigated and the results are summarized as follows:

(1) Er element uniformly distributes in the cell wall matrix in the forms of Al_3Er phase (mainly distributes along the grain boundaries) and Al-Er solid solutions (mainly distribute in the α-Al).

(2) Er element has an important effect on the micro-hardness, yield strength, mean platform stress and energy absorption capacity of the foams. The reasons are mainly due to the solid solution strengthening effect of Er element and second phase strengthening effect of Al_3Er phase.

(3) In order to guarantee the cost effectiveness and compressive properties of the foams, Er element content should be limited. Adding 0.10wt.%–0.50wt.% Er elements to commercially pure Al foams will be more appropriate.

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The study was financially supported by the National Natural Science Foundation of China (No. 51501053 and 51475138) and Science and Technology Plan Projects of Hebei Province (No.15211026).