

Mechanical properties and microstructures of rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys

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$\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys extruded from their rapidly solidified powders have tensile strength more than 800 MPa and Young's modulus about 100 GPa. The extruded $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy is composed of α -Al, Al_3Ni and a metastable tetragonal Al_3Zr , and the extruded $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloy consists of α -Al, Al_3Ni and equilibrium Al_3Ti . Through investigation on microstructure change of rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys with temperature, it is found that a new tetragonal Al_3Zr phase, together with α -Al and Al_3Ni precipitates from the supersaturated α -Al phase in the rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy at around 603 K and an equilibrium Al_3Ti , together with α -Al and Al_3Ni forms from the supersaturated α -Al phase in the rapidly solidified $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloy at about 523 K. The lattice parameters of the new metastable tetragonal Al_3Zr phase were calculated to be $a = 0.3896$ nm and $c = 0.9006$ nm. Both the metastable tetragonal Al_3Zr and equilibrium Al_3Ti phases keep a nano grain size, less than 50 nm even at 773 K. The existence of the nano scale Al_3Zr , Al_3Ti phases and fine grains of α -Al, Al_3Ni phases is the reason that $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys have the high strength. © 1998 Chapman & Hall

1. Introduction

To enhance mechanical properties of Al-based alloys, Zr and Ti are often added to Al-based alloys. When alloys with Zr and Ti are annealed at temperatures ranging from 573 to 773 K, the optimum mechanical properties can be obtained. Addition of Zr or Ti into Al alloys causes formation of metastable superlattice $\text{L}_{12}\text{-Al}_3\text{Zr}$ or $\text{L}_{12}\text{-Al}_3\text{Ti}$ phase. The lattice constants of $\text{L}_{12}\text{-Al}_3\text{Zr}$ and $\text{L}_{12}\text{-Al}_3\text{Ti}$ phases are very near that of α -Al phase. It is said that there exists coherence at the interface of α -Al phase and the $\text{L}_{12}\text{-Al}_3\text{Zr}$ or $\text{L}_{12}\text{-Al}_3\text{Ti}$ phase so that Zr and Ti can enhance the tensile strength of the Al alloys. Recently, Guo and Ohtera [1] found that when the rapidly solidified binary Al–Zr alloys are annealed within a temperature range of 573–773 K, a new tetragonal phase precipitates from the rapidly solidified Al–Zr alloys. This phase has nano grain size and hardly becomes coarse. The new tetragonal phase may contribute Al alloys to the better mechanical properties. On the other hand, it was found that when Zr and Ti are added into Al–Ni based alloys the extruded alloys made from their rapidly solidified powders show tensile strength more than 800 MPa [2–6], while the binary Al–Ni alloy

with the same solute composition has tensile strength only about 600 MPa. This means that Zr and Ti can markedly enhance the tensile strength of Al–Ni alloys. To clarify the reason that Zr and Ti strengthen Al–Ni alloys, microstructures of rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys were investigated and the microstructure and phase change with temperature is shown in this article.

2. Experimental methods

$\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys were prepared from pure elements and gas atomized into powders in an argon atmosphere. The purity is 99.99 wt % for Al, Ni and 99.9 wt % for Zr and Ti. The atomized powders under 26 μm in diameter were extruded at 673 K to form a bulk with a diameter of about 8 mm at an extrusion ratio of 10:1. The mechanical properties of the extruded bulk alloys were measured by an Instron-type tensile testing apparatus.

To investigate microstructure change with temperature, rapidly solidified ribbons of the alloys were used. By using a single roller melt spinning apparatus, the prealloyed ingots were rapidly solidified into a

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ribbon form at a circumferential speed of 40 ms^{-1} in an argon atmosphere. The thickness of these ribbons is in the range of $20\text{--}30 \mu\text{m}$. Thermal stability of these rapidly solidified ribbons was evaluated by differential scanning calorimetry (DSC) to measure the phase transformation temperature. On the basis of DSC results, the as-quenched ribbons were annealed at the phase transformation temperature. Crystal structures of the as-quenched, annealed ribbons and extruded alloys were examined by X-ray diffraction and transmission electron microscopy (TEM). The microstructural change with increasing temperature was in situ observed using TEM with a heating stage.

3. Mechanical properties of $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys

Mechanical properties of the extruded $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ are shown in Table I. It can be seen from the table that these alloys have tensile strength more than 800 MPa and Young's modulus of about 100 GPa. As we know, ultra-high-strength Duralmin alloy has the highest strength among commercial Al alloys. Its strength is less than 600 MPa. Therefore, the strength of the extruded $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys is about 40% higher than that of the ultra-high-strength Duralmin alloy. On the other hand, Young's modulus of commercial Al alloys is about 70 GPa, while these alloys have Young's modulus as high as about 100 GPa. This is also about 40% higher than the commercial Al alloys. Therefore, we can say that the extruded $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys have very high tensile strength and very large Young's modulus.

4. Microstructure of extruded $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys

According to X-ray diffraction results and TEM observation, atomized $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ powders with diameters less than $26 \mu\text{m}$ are composed of only $\alpha\text{-Al}$ phase. The bulk $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy extruded at 673 K from its atomized powders with diameters less than $26 \mu\text{m}$ is composed of $\alpha\text{-Al}$, Al_3Ni and a metastable tetragonal Al_3Zr , and the bulk $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloy extruded at 673 K consists of $\alpha\text{-Al}$, Al_3Ni and equilibrium Al_3Ti . The average grain size of Al_3Ni in the extruded $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys is about 200 nm and the grain size of Al_3Zr and Al_3Ti is smaller than 50 nm. To clarify how the microstructure and phases of the extruded alloys form from its atomized powders, the

TABLE I Mechanical properties of extruded Al–Ni–Zr and Al–Ni–Ti alloys

Alloys (at %)	Tensile strength (MPa)	Elongation (%)	Young's modulus (GPa)
$\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$	835	3.0	98
$\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$	865	4.0	106

microstructure change of rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ ribbons with temperature was investigated.

5. Microstructure change of rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy with temperature

X-ray diffraction result shows that the rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ ribbon consists of only super-saturated solid solution $\alpha\text{-Al}$ phase. On DSC curve of the ribbon shown in Fig. 1 there is an exothermic peak at 603 K. To determine what kinds of phases form at the temperature corresponding to the exothermic peak and whether there are other phases forming above the temperature, the rapidly solidified ribbons were annealed at 603, 673, 773 and 873 K for 3.6 ks, and their phase structures were analyzed by X-ray diffraction. X-ray diffraction results shown in Fig. 2 prove that the precipitating phases at 603 K are Al_3Ni and a new metastable tetragonal Al_3Zr phase, and above 603 K there is no other phase forming. The lattice parameters of the new metastable tetragonal

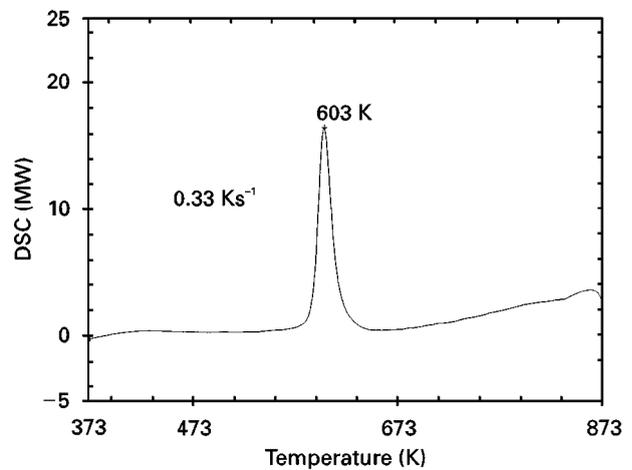


Figure 1 DSC curve of rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy.

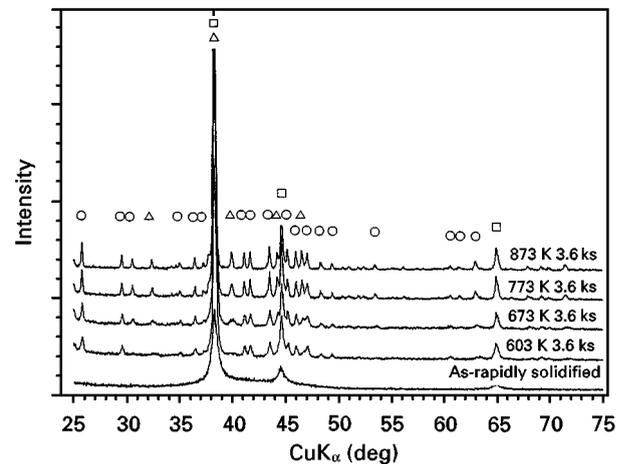


Figure 2 X-ray diffraction patterns of $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy. (□) $\alpha\text{-Al}$; (○) Al_3Ni ; (△) Al_3Zr metastable tetragonal phase. $a = 0.3896 \text{ nm}$, $c = 0.9006 \text{ nm}$.

Al_3Zr phase were calculated to be $a = 0.3896$ nm and $c = 0.9006$ nm.

The microstructure change with increasing temperature was observed *in situ* using TEM with a heating stage when the $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ ribbon was heated. As shown in Fig. 3a, the microstructure of as-rapidly solidified ribbon is very fine. After the ribbon was heated at 603 K, many fine particles precipitated from α -Al phase (Fig. 3b). As temperature rose, some of the precipitated particles grew much larger and some of them became a little bigger (Fig. 3c–f). It is confirmed by selected area and nano beam diffraction spots of TEM that the larger grains are Al_3Ni and the very fine grains with size less than 50 nm are the metastable Al_3Zr phase.

6. Microstructure change of rapidly solidified $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloy with temperature

As shown in Fig. 4, rapidly solidified $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ ribbon is also composed of only supersaturated α -Al phase. On DSC curve of the ribbon, there is an exothermic peak at 523 K. To determine what kinds of phases precipitate from α -Al phase, the rapidly solidified ribbons were annealed at 523, 573, 673, 773 and 873 K for 3.6 ks and the phases were identified by X-ray diffraction. The X-ray diffraction results shown in Fig. 4 prove that Al_3Ni and equilibrium DO22- Al_3Ti phases precipitate from the supersaturated α -Al phase at 523 K. No other phase appears at temperatures above 523 K.

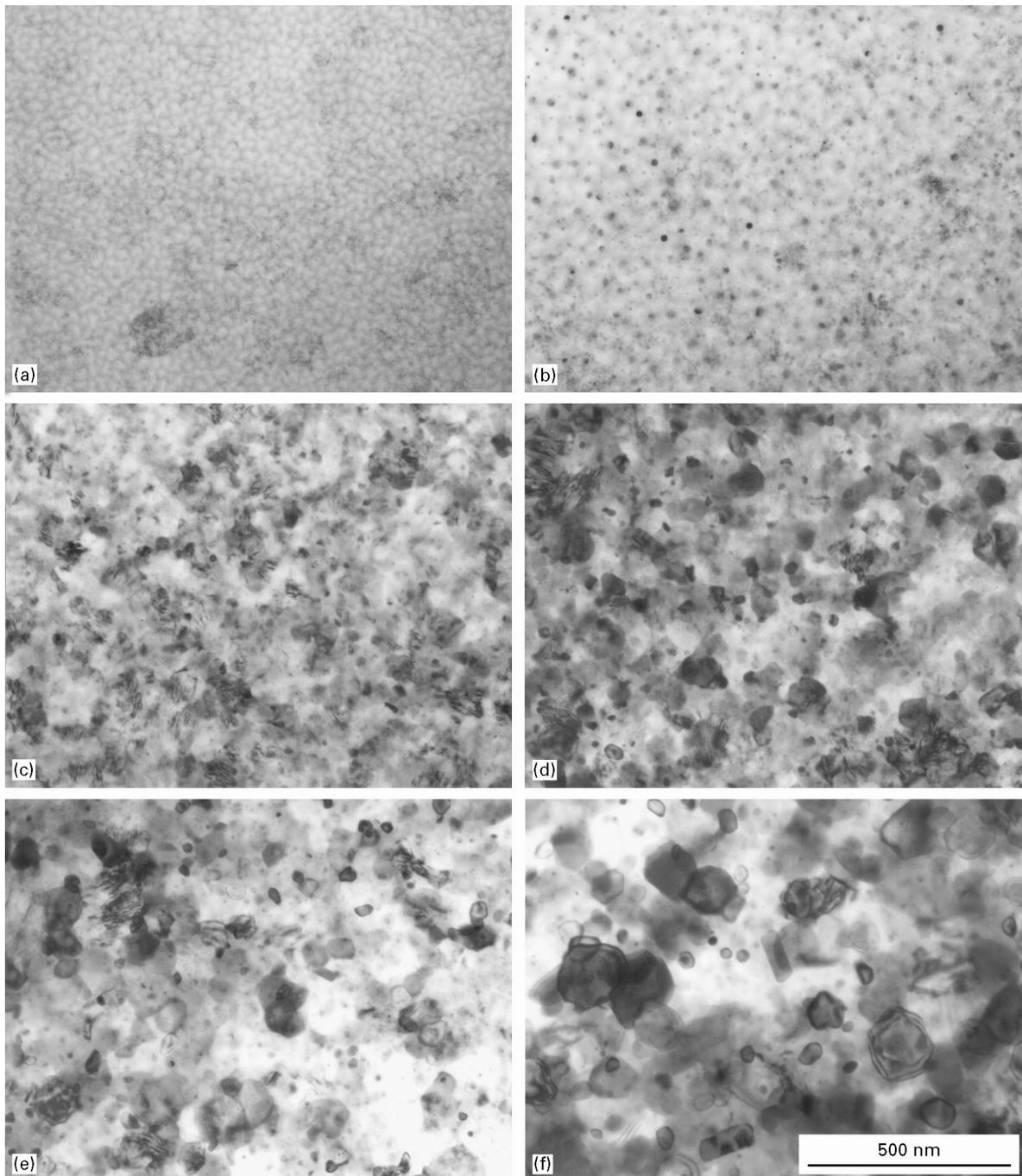


Figure 3 Microstructural change of rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy with increasing temperature. (a) As-rapidly solidified; (b) 603 K, 900 s; (c) 623 K, 900 s; (d) 673 K, 900 s; (e) 723 K, 900 s; (f) 773 K, 900 s.

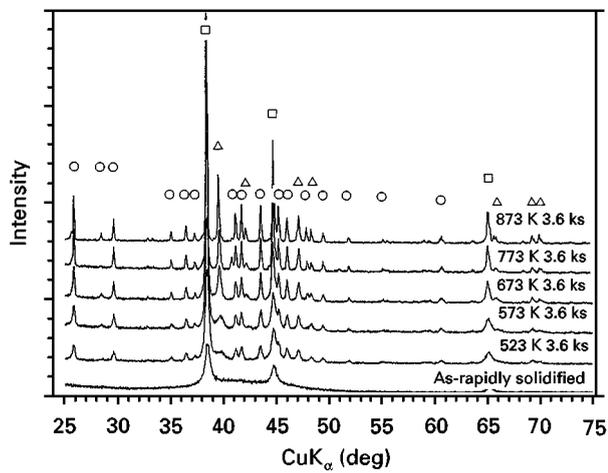


Figure 4 X-ray diffraction patterns of $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloy. (\square) α -Al; (\circ) Al_3Ni ; (\triangle) $\text{DO}_{22}\text{-Al}_3\text{Ti}$.

The microstructure change with increasing temperature is shown in Fig. 5. It can be seen from Fig. 5a that the supersaturated α -Al grains are very large in as-rapidly solidified ribbons. This is different from the microstructure of as-rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy shown in Fig. 3a. The precipitates from the phase at 573 K are fine (Fig. 5b). As temperature rises, the Al_3Ni phase begins coarsening. On the contrary, the grain size of Al_3Ti is very fine, i.e. smaller than 50 nm.

7. Discussion

On the basis of above results it is known that after annealing at high temperature the rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy decomposes into α -Al, Al_3Ni and a metastable tetragonal Al_3Zr at 603 K, and the

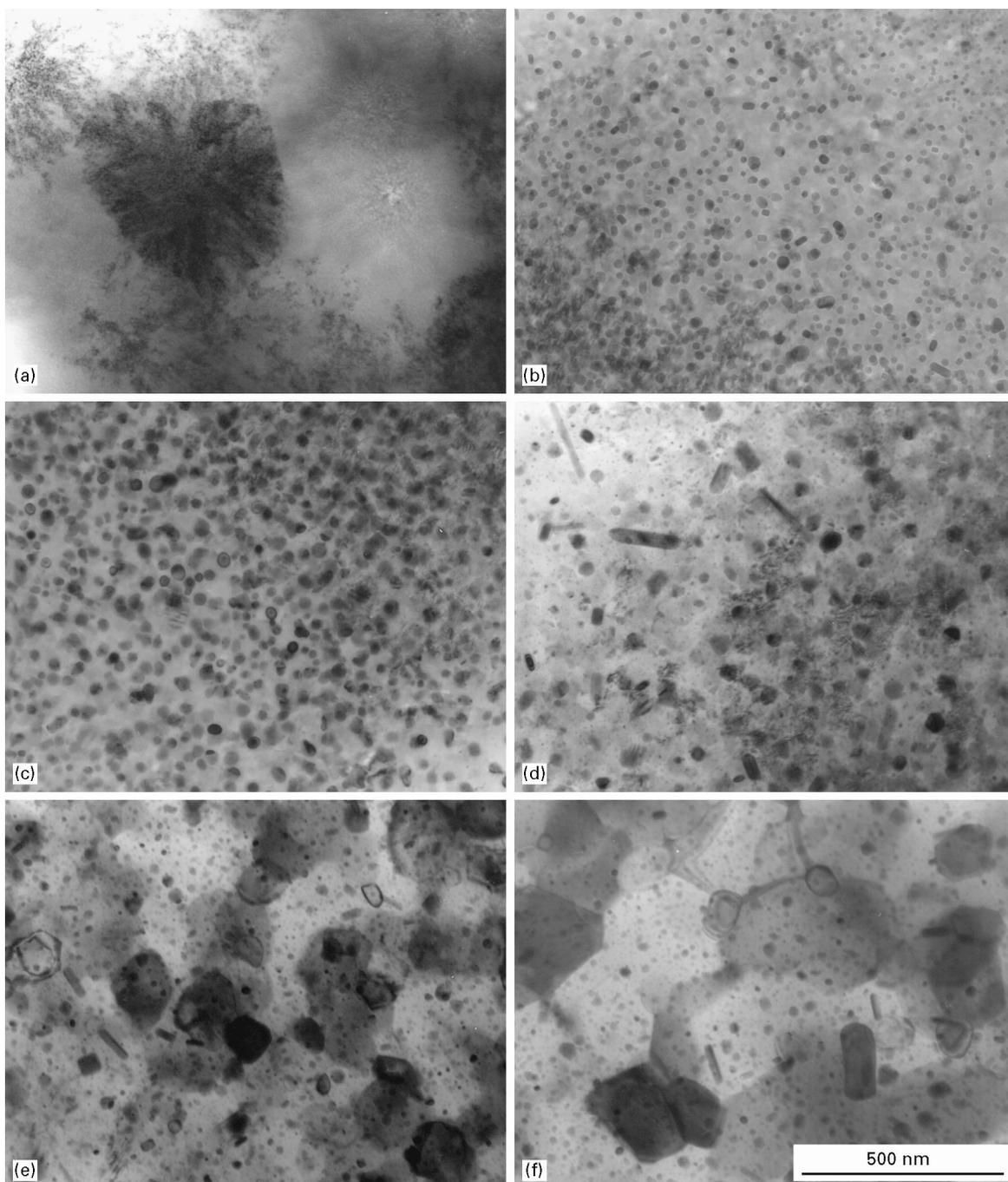


Figure 5 Microstructural change of rapidly solidified $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloy with increasing temperature. (a) As-rapidly solidified; (b) 573 K, 900 s; (c) 623 K, 900 s; (d) 673 K, 900 s; (e) 723 K, 900 s; (f) 773 K, 900 s.

rapidly solidified $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloy transforms to $\alpha\text{-Al}$, Al_3Ni and equilibrium Al_3Ti at 523 K. At higher temperature the grains of the Al_3Ni phase become coarse, while the metastable tetragonal Al_3Zr and equilibrium Al_3Ti phases keep a nano grain size. The phases appearing in the atomized powders and extruded bulk are the same as those in the ribbons as-rapidly solidified and annealed at 673 K, respectively. On the other hand, metastable ordered $\text{L}_{12}\text{-Al}_3\text{Zr}$ and $\text{L}_{12}\text{-Al}_3\text{Ti}$ phases are not found to exist in these alloys. Further, in rapidly solidified binary Al-Zr alloys the ordered $\text{L}_{12}\text{-Al}_3\text{Zr}$ phase was observed to transform into the metastable tetragonal Al_3Zr in a temperature range of 573–773 K [1]. So, although the $\text{L}_{12}\text{-Al}_3\text{Zr}$ phase can form in rapidly solidified Al-based alloys, it will change to the metastable tetragonal Al_3Zr phase at temperatures higher than 573 K. Therefore, the existence of nano scale tetragonal Al_3Zr and the equilibrium Al_3Ti phase in $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys is an important reason that these alloys have very high tensile strength. The fine grains of $\alpha\text{-Al}$ and Al_3Ni phases, together with nano scale Al_3Zr and Al_3Ti phases contribute to the high strength and the Young's modulus of the alloys.

8. Conclusions

$\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ and $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloys extruded from their atomized powders with diameters less than 26 μm have tensile strength more than 800 MPa and

Young's modulus of about 100 GPa. After annealing at 603 K, the rapidly solidified $\text{Al}_{89.5}\text{Ni}_8\text{Zr}_{2.5}$ alloy decomposes into $\alpha\text{-Al}$, Al_3Ni and a metastable tetragonal Al_3Zr phase with lattice parameters of $a = 0.3896 \text{ nm}$ and $c = 0.9006 \text{ nm}$, and the rapidly solidified $\text{Al}_{88.5}\text{Ni}_8\text{Ti}_{3.5}$ alloy transforms to $\alpha\text{-Al}$, Al_3Ni and equilibrium Al_3Ti at 523 K. At higher temperatures the grains of the Al_3Ni phase become coarse, while the metastable tetragonal Al_3Zr and equilibrium Al_3Ti phases keep a nano grain size. The fine grains of $\alpha\text{-Al}$, Al_3Ni phases, together with nano scale Al_3Zr and Al_3Ti phases contribute to the high strength and the Young's modulus of the alloys.

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Received 1 May

and accepted 24 September 1997