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Thermal Stability, Formability, and Mechanical Properties of a High-Strength Rolled Flame-Resistant Magnesium Alloy

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Additional information is available at the end of the chapter

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1. Introduction

Lightweight magnesium (Mg) alloys with excellent shock-absorption properties are being actively adopted for electronic information devices and automotive parts [1]. Mg alloys shows excellent environmental properties because of their light weight, which can lead to improved energy efficiency and hence a reduction in emissions of carbon dioxide [1–2]. For use in structural applications, Mg alloys need to have adequate ductility, thermal stability, and strength. However, Mg alloys often exhibit low ductility, low tensile yield strength, and poor formability as a result of limited slip in their hexagonal close-packed structures [2]. The known ways for effectively improving the mechanical properties and formability of Mg alloys include grain refinement [3–4] and control of the texture of the alloy [4–5]; both these techniques promote prismatic slip and facilitate the creation of large plastic deformations. It is well known that the ignition temperature of Mg alloys is lower than that of other materials for vehicles [6]. However, the ignition temperature of Mg alloys can be markedly raised by the presence of small amounts of calcium (Ca) [6]. Recently, AZX and AMX (X = Ca) alloys have been shown to have higher ignition temperatures than other Mg alloys [6–7]. The effect of adding Ca to improve the flame resistance of Mg alloys has been demonstrated experimentally, as shown in Figure 1. At a temperature at 550 °C, AZ61 Mg alloy ignites, the surface of A6N01 alloy begins to change color, and AZX611 alloy is unaffected.

In general, Mg alloys that contain other elements are known to have greatly enhanced mechanical properties, but their ductility can be maintained only processing the cast metal through extrusion and/or plastic deformation [10–11]. It has been suggested that grain

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refinement of the Mg phase occurs during extrusion deformation [3,10–12]. The strength and microstructure of extruded Mg–Ca and Mg–Ca–Al(Zn) [6–7,9–13] alloys have been examined, together with the distribution of compounds within these materials [7,12,15]. Most of the plastic deformation processes to which Ca-containing Mg alloys have been subjected are extrusion processes [9–10], and there have been few attempts to examine improvements in the strength of these materials by means of rolling and working processes. It is widely known that the plastic deformation of alloys of Mg with rare earth (RE) metals [8] or Ca [11–12] requires many working cycles and high processing temperatures in comparison with commercial Mg alloys [5,7]. If wrought Mg materials are to be more widely used, it will be important to develop techniques for the production of rolled sheets in addition to extruded materials. It will also be necessary to elucidate the mechanical properties of such materials and to establish a rolling process for Mg alloys that is faster than the current extrusion processes. It has been reported that a rolling process has been carried out at processing temperatures (sample temperature, roll surface temperature, and reheating temperature) above the static recrystallization temperature [6–13]. However, there have been no previous studies on the relationship between the microstructural changes responsible for strength enhancement and rolling processes without reheating or of the maximum reduction in thickness achievable in single-pass rolling.

In this study, we investigated the changes in the microstructure and tensile properties at room temperature produced by controlled rolling of samples of Mg–Al–Mn–Ca cast alloy produced by a twin-roll casting process. The heat resistance, formability, and damping properties of the rolled sheet produced were compared with those of the A6N01 alloy currently used in high-speed rail vehicles [14].
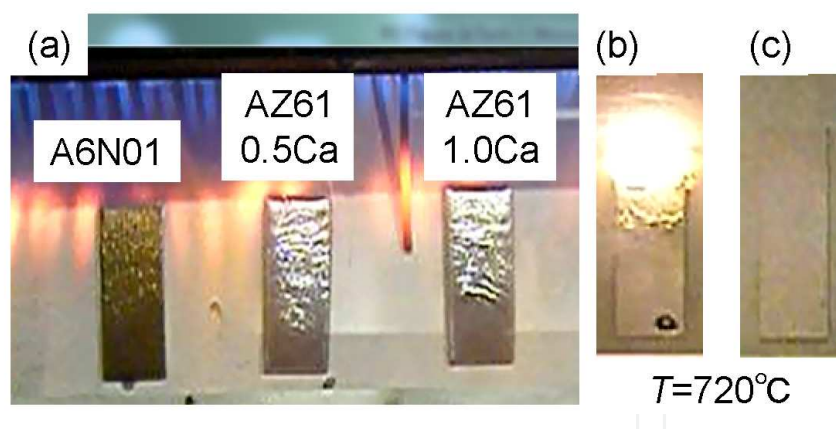

Figure 1. Appearance of samples of A6N01, AZ61+0.5Ca and AZ61+1Ca alloys subjected to flame-resistance testing (a). Ignition temperature of Ca addition magnesium AZX611 alloy (c) higher than AZ61 (b) magnesium alloys.

2. Experimental procedure

The alloy used in this study was twin roll cast (TRC) Mg–10Al–0.2Mn–1Ca (mass%) alloy (AMX1001). The material properties of this alloy were compared with those of samples of

Mg–3Al–1Zn–1Ca (AZX311) alloy and Mg–6Al–1Zn–1Ca (AZX611) alloy. Ingots were prepared in an electric furnace in an atmosphere of argon. The as-received samples of the TRC material measured 100 mm wide by 2000 mm long by 4 mm thick. Samples for rolling, measuring 60 mm wide and 120 mm long, were cut from the as-received materials. The TRC direction and rolling direction were parallel to one another, and the microstructure was observed from the direction perpendicular to the direction of rolling and casting. Rolling was carried out on samples preheated to 100 to 400 °C for 10 min in a furnace. The surface temperature of the rollers was maintained at 250 °C by means of embedded heating elements. The sample was rolled from a thickness of 4 mm to one of 1 mm in several passes (1 mm per pass) without reheating during the rolling process, or by single-pass rolling. The roll diameter was 180 mm and roll speed was set at 5, 10, 15, or 25 m/min. We chose to roll the sheets to a thickness of 1 mm to permit comparison of the mechanical properties of sheets produced by multipass rolling with those of sheets rolled in a single pass. The samples were cooled with water within 5 s of the final rolling pass. The AMX1001 alloy consisted of an α-Mg phase and Al–Ca compounds [13].

Samples for tensile testing with gauge sections 2.5 mm in width and 15 mm in length were machined from samples of the rolled and annealed materials. Tensile tests were carried out at an initial strain rate of 5.0×10^{-4} s^{-1} at room temperature. Samples were annealed at tempera-tures of between 100 and 400 °C in an electric furnace for various times between 1 and 1000 h, with subsequent cooling in water. The formability of the rolled sheets was investigated by conical cup tests performed temperatures between room temperature and 250 °C at an initial strain rate of 2.7×10^{1} s^{-1}. The microstructures of the rolled and annealed samples were observed by optical microscopy (OM), scanning electron microscopy (SEM), and electron backscattering diffraction (EBSD). The optical and Electron probe micro-analyser (EPMA) maps of as-cast AMX1001 alloy are shown in Figure 2. The initial mean grain size of AMX1001 cast alloy was 53 μm. The brighter areas in Figure 2 correspond to Al$_2$Ca compounds. The Al$_2$Ca compounds were present as networks and as coarse agglomerations.
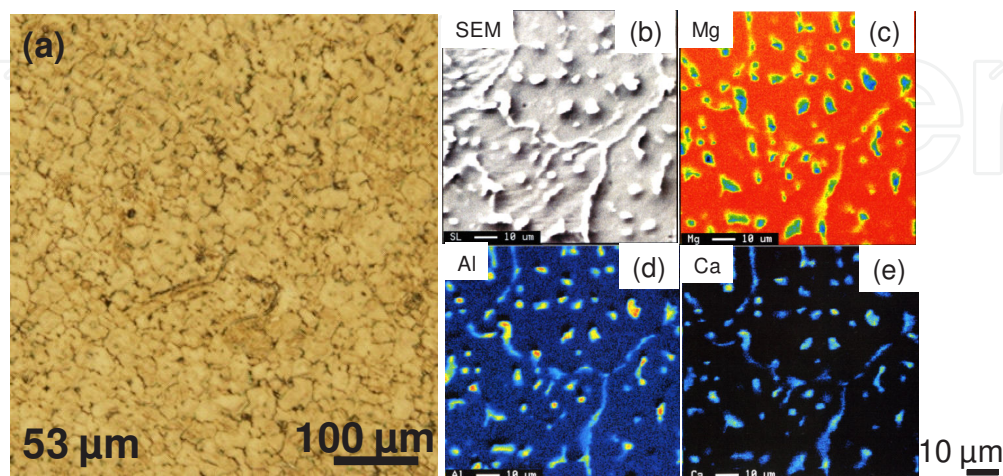

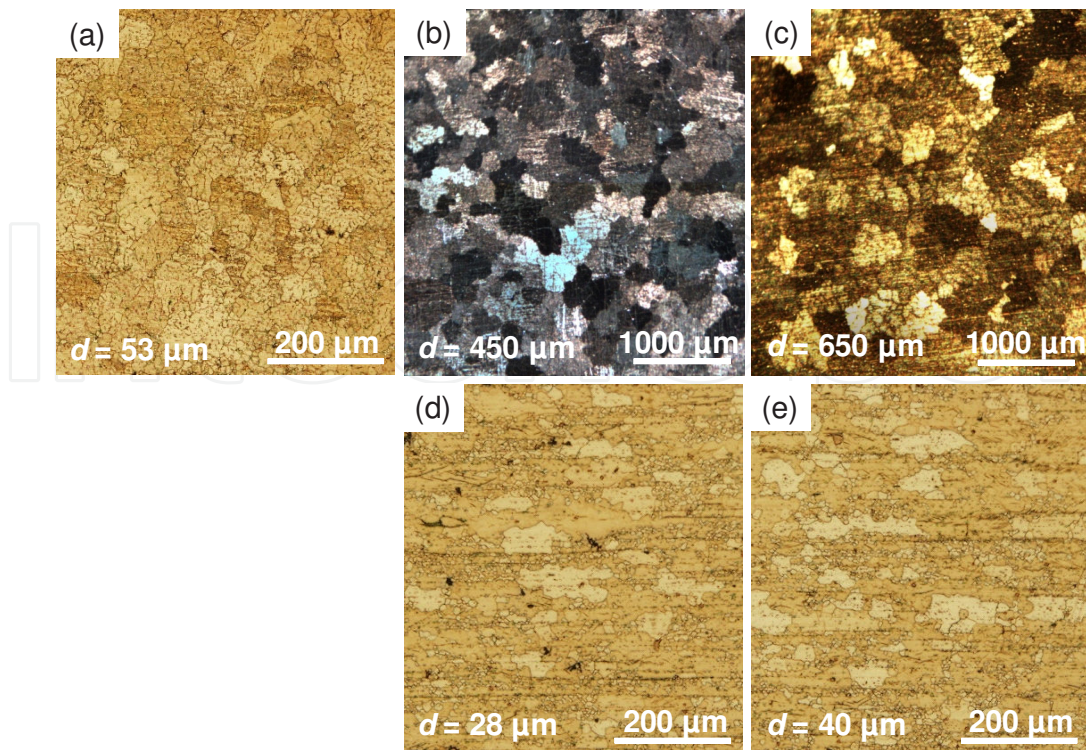**Figure 2.** Optical micrograph (a) and EPMA maps (b)-(e) of cast sample of AMX1001 twin roll cast alloy.

Figure 3. Optical microstructures of twin rolled cast alloy AMX1001 (a), gravity-cast alloys AZX311 (b) and AZX611 (c), and extruded alloys AZX311 (d) and AZX611 (e).

3. Results and discussion

3.1. Structure of the cast materials and the limited reduction in thickness diagram

Rapidly cooled AMX1001 TRC alloy (the as-received material) has a finer grain structure than AZX311 or AZX611 gravity-cast alloys. However, the mean grain size of the AZX311 and AZX611 alloys was similar to that of AMX1001 TRC. The initial optical microstructure of these materials is shown in Figure 3. It is necessary to know the relationship between the sample temperature and the maximal reduction in thickness per pass in order to prepare thin sheets without cracking. Figure 4(a) shows the maximum reduction in thickness per pass in strip pressing of samples of AZX311, AZX611, and AMX1001 cast alloy; optical micrographs of samples of AZX311 and AMX1001 subjected to single-pass rolling at 200 °C are shown in Figure 4(b) and (c).

The maximum reduction in thickness at a deformation temperature of 200 °C for samples of AZX311 and AZX611 cast alloys was 9%, whereas that of AMX1001 cast alloy was 30% [Figure 4(a)]. This shows that AMX1001 TRC alloy has a higher deformability than AZX311 and AZX611 cast alloys. Furthermore, the maximum reduction in thickness reached 50% for AMX1001 TRC alloy when the deformation temperature was raised to 300 °C; the maximum reduction in thickness therefore increases significantly at deformation temperatures between

200 and 300 °C. Optical micrographs recorded after applying a reduction in thickness of 20% to AMX1001 TRC alloy at a sample temperature at 200 °C and a roll surface temperature of 250 °C are shown in Figure 4(b) and (c). It can be seen that, in comparison with the cast material, shear deformation is introduced into the microstructure. As will be discussed later, dynamic recrystallization (DRX) occurs on elevating the sample temperature after the rolling process. As a result of the rolling process, the α-Mg phase becomes finer, Al–Ca compounds are finely crushed, and structural rearrangement occur; furthermore, the Mg phase and the Al–Ca compounds exhibit a lamellar structure.
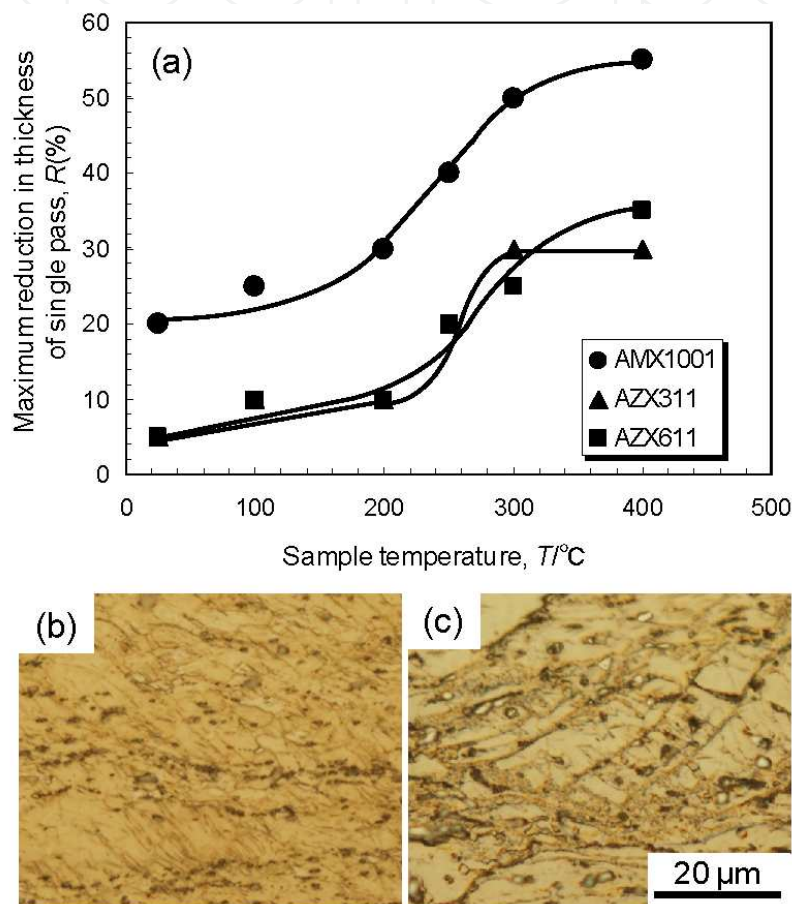

Figure 4. Maximum reduction in thickness curves for AZX311, AZX611, and AMX1001 cast alloy (a) and optical micrographs of AZX311 (b) and AMX1001 (c) alloys rolled at 200 °C in a single pass.

Curves showing the maximum reduction in thickness of extruded AZX311 and AZX611 alloys are presented in Figure 5 [7]. The maximum reduction in thickness of AMX1001 cast material was the same as that of extruded AZX311 and AZX611 alloys. In other words, the TRC material shows a good rollability in comparison with the gravity-cast materials when the mean grain size is less than 100 μm. The TRC process, which involves rapid cooling, therefore has advantages in terms of the rollability of the product, in addition to its greater productivity. By the way, Figure 5 shows that the maximum reduction in thickness of AZX611 alloy decreases when the deformation temperature is increased to more than 400 °C. This decrease is probably

the result of growth of grains of α-Mg phase and the proximity of the annealing temperature to the solution-treatment temperature [13]. These observations are consistent with the forging and molding properties of AZ31 alloys [15] and Mg–Zn–Y alloys [8], and also with the fact that AZ31 alloy is a nonaging alloy, whereas AZ61 alloy is an aging alloy. In other words, the roll surface temperature and the reduction in thickness are important factors in strip processing of AMX1001, AZX311, and AZX611 alloys, and a fine dispersion of Al_2Ca compounds occurs as a result of the refinement of the α-Mg phase.
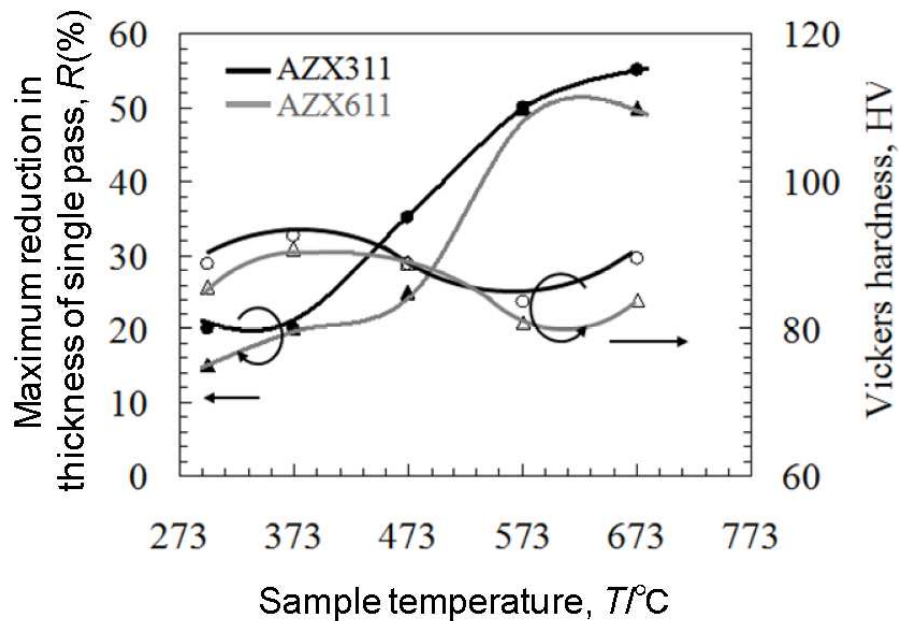

Figure 5. Maximum reduction in thickness curves for AZX311 and AZX611 extruded alloy at 200 °C after a single pass. The results for AMX1001 cast alloys are also shown in this figure [7].

3.2. Effects of rolling conditions on the microstructure of flame-resistant Mg alloy

To investigate the effect of rolling conditions and grain refinement, we used 10-mm-thick samples of AZX611 gravity-cast material with a coarse grain, because increasing the total reduction in thickness eliminated the fine structure of the cast material.

Figure 6(a) shows the tensile properties of the as-cast alloys subjected to rolling at a sample temperature of 200 °C or 400 °C with a roll surface temperature of 250 °C. Samples were rolled from a thickness of 10 to 1 mm in nine passes at 200 °C (the 200 °C rolled sheet) or in a single pass at 400 °C (the 400 °C rolled sheet). The yield stress (YS) of the 200 °C rolled sheet was 340 MPa, its ultimate tensile strength (UTS) was 350 MPa, and its elongation was 4%. The YS and UTS of the 400 °C rolled sheet were 220 and 270 MPa, respectively, and its elongation was 11%. In the single-pass rolling at a sample temperature of 400 °C, we were able to reduce the thickness by 90%, because the sample temperature was close to the solution temperature and the Mg phase and the Al–Ca compounds could be easily deformed. The YS and UTS of the resulting sample were lower than those of the alloy sample rolled at 200 °C, but the elongation

was improved. The difference between the temperature of the sample (200 °C) and the roll surface temperature (250 °C) suppresses loss of heat during the rolling process, thereby inducing DRX and increasing plastic deformability. Optical micrographs of the 200 °C and 400 °C rolled materials are shown in Figure 6(b) and (c). The Al–Ca compounds are finely crushed and dispersed during the rolling process, and fine Al–Ca compounds are formed at grain boundaries. It is well known that finely crushing Al–Ca compounds contributes to control of grain growth [7]. A comparison of the textures in optical micrographs of the 200 °C rolled sheet after nine passes and in the 400 °C rolled sheet after a single pass showed that the former contained relatively equiaxial grains, whereas the latter showed a microcrystalline structure between the grain boundaries and a completely uniform texture was not formed. Therefore, strip processing for a few passes under a small load to produce a uniform texture is effective in reducing the total load and increasing the strength of the alloy without reheating.
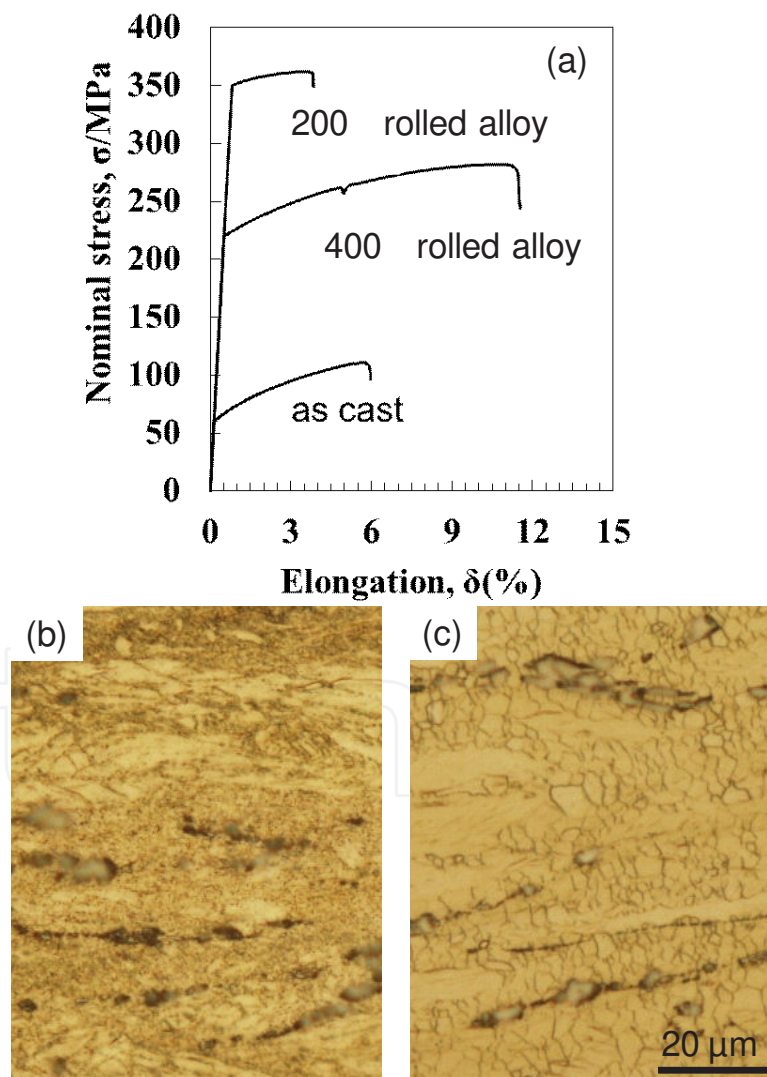

Figure 6. Nominal stress–strain curves for AZX 611 sheet rolled at 200 °C (multipass) or 400 °C (single pass) (a), and optical micrographs of the materials rolled at 200 °C (b) and 400 °C (c).

Figures 7(a–f) show the inverse pole figure (IPF) and pole figure (PF) maps of alloy samples rolled at 200 °C [Figures 7(a–c)] or 400 °C [Figures 7(d–f)]. In the sample rolled at 200 °C, the recrystallized region amounted to 81% of the rolled sheet, and the mean grain size was reduced from 1140 μm to 3.5 μm (the mean grain sizes in the recrystallized and the nonrecrystallized regions were 1.5 μm and 9.4 μm, respectively) (Figure 7a). The sample and roll surface temperatures of 200 °C and 250 °C, respectively, are close to the static and dynamic recrystallization temperatures of Mg alloy [10]. The microstructure was refined by DRX, resulting in a duplex grain structure consisting of partially elongated grains, and shear deformation was observed. As can be seen in Figs. 7(b–c), the intensity of the basal texture of the AZX611 rolled sheet was 10.2; the intensities of the basal textures of the recrystallized and nonrecrystallized regions were 9.8 and 19.6, respectively. As shown in Figures 7(d–f), the sample rolled at 400 °C contained a region with elongated grains and fine grains. The recrystallized region of the rolled sheet accounted for 47% of the total, and the mean grain sizes of the recrystallized and nonrecrystallized regions were 2.9 and 27.8 μm, respectively. As can be seen in Figures 7(a–c), the intensity of the basal texture of the AZX611 rolled sheet was 13.9, and the intensities of the basal textures of the recrystallized and nonrecrystallized regions were 4.5 and 21.4, respectively. The sample temperature of 400 °C is near the grain growth and solution temperature of AZX611 and AZ61 alloys [7,12–13]. The microstructure was refined by DRX, but grain growth occurred immediately after rolling. As a result, the alloy rolled at 400 °C showed a duplex grain structure, the intensity of the basal texture of the nonrecrystallized region was higher than that for the sample rolled at 200 °C, and the area frequency of the nonrecrystallized region was more than 50%. Therefore, if there is a difference in the sample temperature, but no observed significant differences in the mean grain size, provided the nonrecrystallized region is taken into consideration, the grain-refinement mechanism is dependent on the sample temperature and the reduction in thickness.

Next, we focused on the sample temperature after rolling in the nine-pass process that produced high-strength AZX611 rolled sheet. Figure 8(a) shows the results of measurements of the sample temperature after rolling and the mechanical properties for various total reductions in thickness. Figures 8(b–d) show optical micrographs for the several reductions in thickness, as indicated in Figure 8(a). From Figure 8(a), it is apparent that when the total reduction in thickness was less than 60%, the sample temperature after rolling increased slightly as the number of rolling passes increased. On the other hand, the sample temperature after the rolling process did not show any increase for a total reduction in thickness of more than 60%. The strength increased after a total reduction in thickness of 60%, but the elongation was reduced. Figures 8(b–d) show that the dendrite structure was also elongated in the rolling direction after a total reduction in thickness of 40% and that Al–Ca compounds remained in the grain boundary. The existence of shear deformation of the microstructure when the total reduction in thickness reached 60% was confirmed and, at this stage, Al–Ca compounds were beginning to be crushed. When the total reduction in thickness reached 80%, bending of the microstructure occurred as a result of shear deformation and elongation of the grain in the direction parallel to the rolling direction. By performing multipass rolling and introducing shear deformation by the rolling process, we were able to increase the strength of the Mg phase by DRX, while the Al–Ca compounds were crushed. This was possible because the deformation
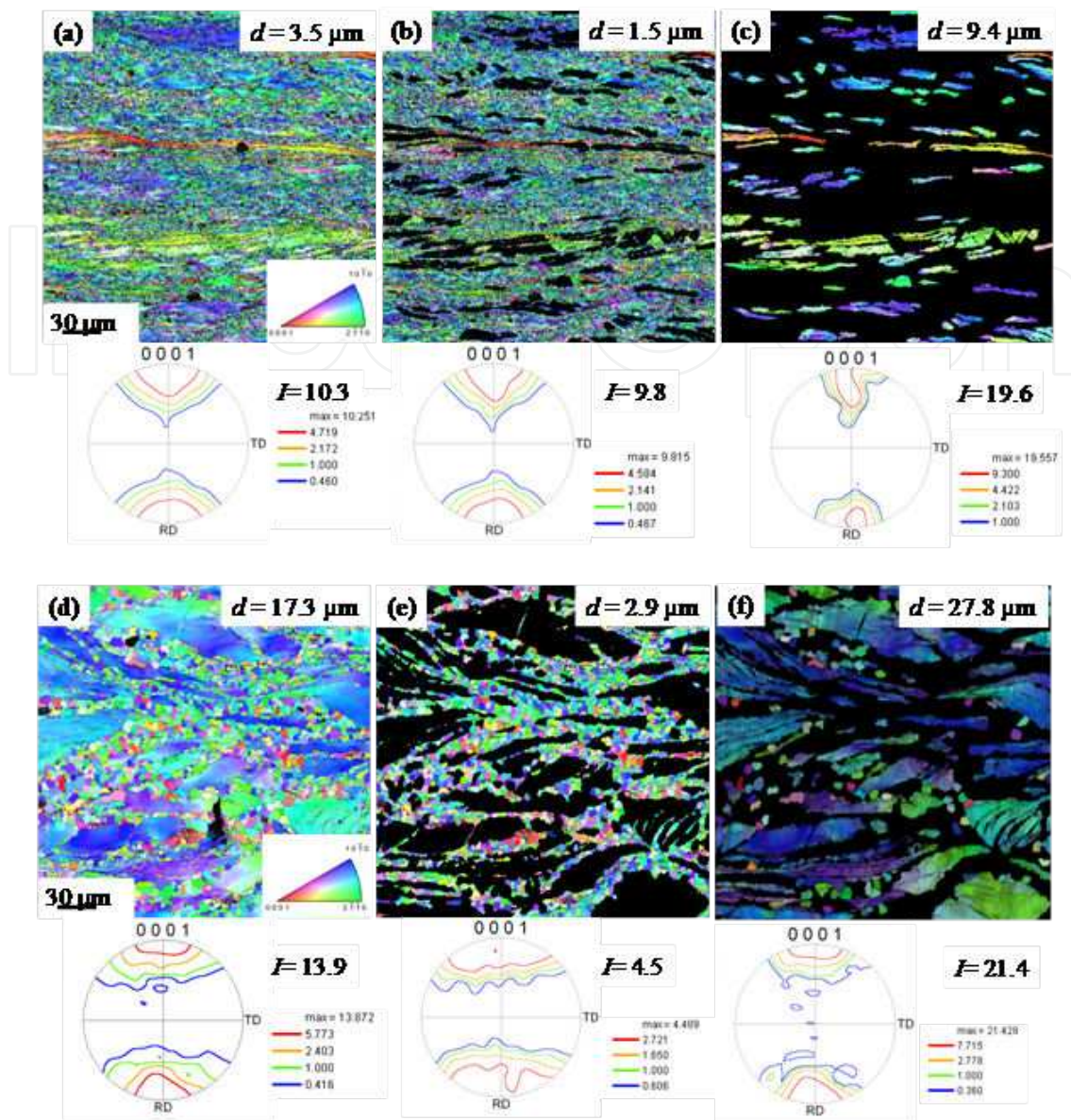
Figure 7. Inverse pole figure and pole figure maps of samples of AZX611 alloy rolled at 200 °C (a)–(c) or 400 °C (d)–(f); (b) and (e) show the recrystallized regions and (c) and (f) show the nonrecrystallized regions.

process induced heating of the alloy during each rolling pass, thereby maintaining the sample at a temperature near its solution temperature. Al–Ca compounds that had been crushed were rearranged in layers in the Mg phase in the direction parallel to the rolling direction.

3.3. Production of high-strength Mg–10Al–0.2Mn–1Ca rolled sheet by a rolling process

From Figures 2 and 3, the initial grain size of AMX1001 TRC alloy was lower, by a ratio of 1:20, than the mean grain size of AZX311 and AZX611 gravity-cast alloy, so that improved rollability would be expected. Therefore, a sample of AMX1001 was rolled from a thickness of 4 mm to 1 mm in three passes. Figures 9(a–c) show the relationship between the mechanical properties and various rolling conditions for AZX1001 alloy. Figure 9(a) shows that, as a result of the
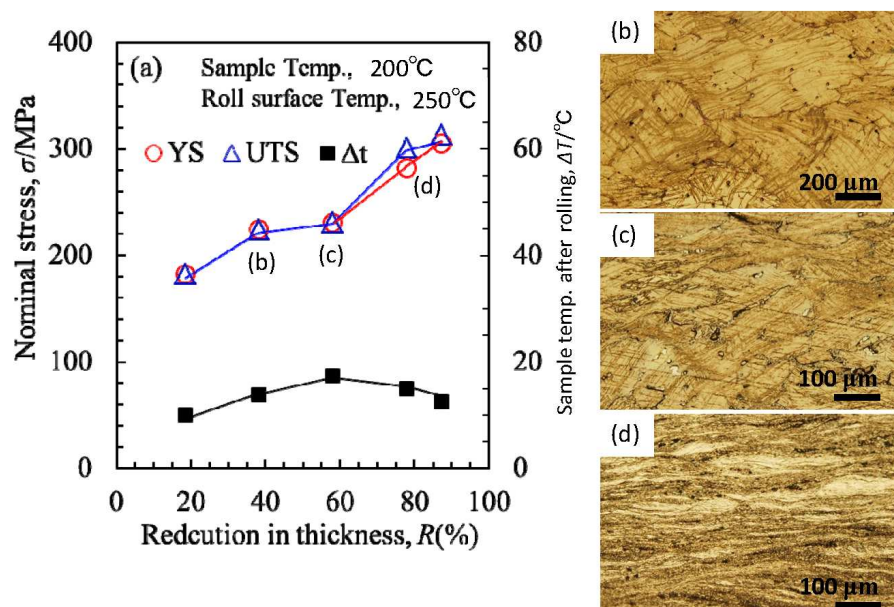
Figure 8. Relationship between sample temperature after the rolling process and the mechanical properties of AZX611 (a); optical micrographs for samples of AZX611 reduced in thickness by 40% (b), 60% (c), and 80% (d).

multipass rolling process, the YS decreased slightly from 390 to 340 MPa and UTS also decreased slightly from 410 to 380 MPa, probably due to the increase in sample temperature from 100 to 350 °C, whereas the elongation improved from 3.5 to 8.5%. Additionally, it was clear that the YS and UTS of samples subjected to single-pass rolling were lower than those of samples subjected to multipass rolling process, but the single-pass rolling markedly improved the elongation to 20% [Figure 9(b)]. These results suggest that the elongation of rolled materials depends on the sample temperature, whereas the YS and UTS depend on the number of rolling passes. The multipass rolling regime that we used did not involve reheating between individual passes, and we suggest that recovery and DRX do not act effectively during the initial passes of the rolling process. From Figures 7 and 8, it is clear, however, that recovery and DRX do act effectively when the reduction in thickness reaches more than 70%. Figure 9(c) shows that the YS and UTS decrease with increasing rolling speed for a total reduction in thickness of 80%, even with a multipass rolling process. This is because there is an increase in the sample temperature after rolling, as in the case of extrusion [8]. It is possible to combine single- and multipass rolling processes to suit particular applications; the rolling speed and sample temperature are the important factors in the rolling process. In addition, a total reduction ratio of 60% or more is necessary to produce high-strength rolled material.

Figures 10(a–c) show optical micrographs of the as-cast sample and samples of rolled sheet subjected to rolling for three passes, together with the corresponding IPF and PF maps for a sheet rolled at a speed of 10 m/min at a sample temperature of 200 °C and a roll surface temperature of 250 °C. The microstructure of the cast ingot consisted of a coarse grain structure with Al_2Ca and Al compounds. After the three-pass rolling process, the recrystallized region of the rolled sheet accounted for 70% of the total, and the grain size was reduced from 53 to 3.8 μm. The rolling temperature of 200 °C is close to the recrystallization temperature of both
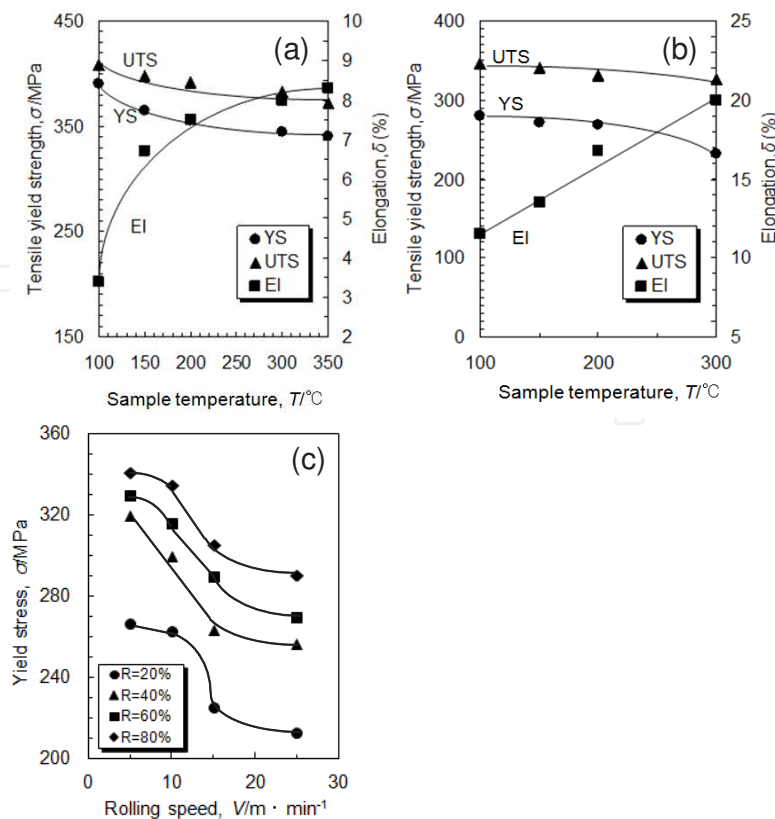
Figure 9. Relationship between the mechanical properties and rolling conditions for a multipass process (a) and a single-pass process (b) performed at various sample temperatures. Relationship between mechanical properties and rolling speed at a sample temperature of 200 °C and roll surface temperature at 250 °C for a reduction in thickness of 80% (c).

AZX and AZ series alloys [11–12,15]. The microstructure was refined by DRX to form a duplex grain structure that was partially elongated, and shear deformation was observed into the elongated grain. To increase the extent of the recrystallized region, an increase in the rolling temperature or a greater total reduction thickness was required, but the strength then tended to decrease because of the influence of grain growth. The Al–Ca compounds were finely crushed and dispersed during the rolling process, and therefore these compounds contributed to control of the grain growth that would otherwise result from an increase in the rolling temperature. As can be seen in Figure 10(c), the intensity of the basal texture of the AMX1001 rolled sheet was 8.2. This basal texture was lower than those of AZ31 alloy samples subjected a single rolling process at 200 or 400 °C, until the rolling reduction reached 86%, at which point the intensities were 7 and 5, respectively [16]. Figure 11 shows the tensile properties of the as-cast and rolled (single- and three-pass schedules) alloys for a roll surface temperature of 250 °C and a rolling speed of 10 m/min; multipass rolling was performed at 100 °C, and single-pass rolling was performed at a sample temperature of 400 °C. The YS and UTS of the sheet subjected to three-pass rolling were 380 and 400 MPa, respectively, and the elongation was 8%. A sheet rolled from a thickness of 4 mm to one of 1 mm in a single pass showed a UTS of 320 MPa, a YS of 220 MPa, and an elongation of 15%. The failure to improve the strength of

the material by the three-pass rolling process was the result of the heat generated during the metal-forming process. In fabricating the 1-mm-thick rolled sheet by the three-pass rolling process, grain refinement of the Mg phase and crushing of the Al-Ca compounds occurred. The difference between the temperature of the sample and that of the roll suppresses heat removal during the rolling process, thereby inducing DRX and increasing deformability.

If no reheating is incorporated into the rolling process, the sample and roll-surface temperatures are close to the recrystallization temperature of the Mg alloy [7,15]. As a result, it is possible to induce DRX through the increase in sample temperature generated by repeated rolling. As a result of the temperature difference between the sample and roll surface, the sample temperature approaches the roll surface temperature more closely as the number of passes increases, making this an important factor.
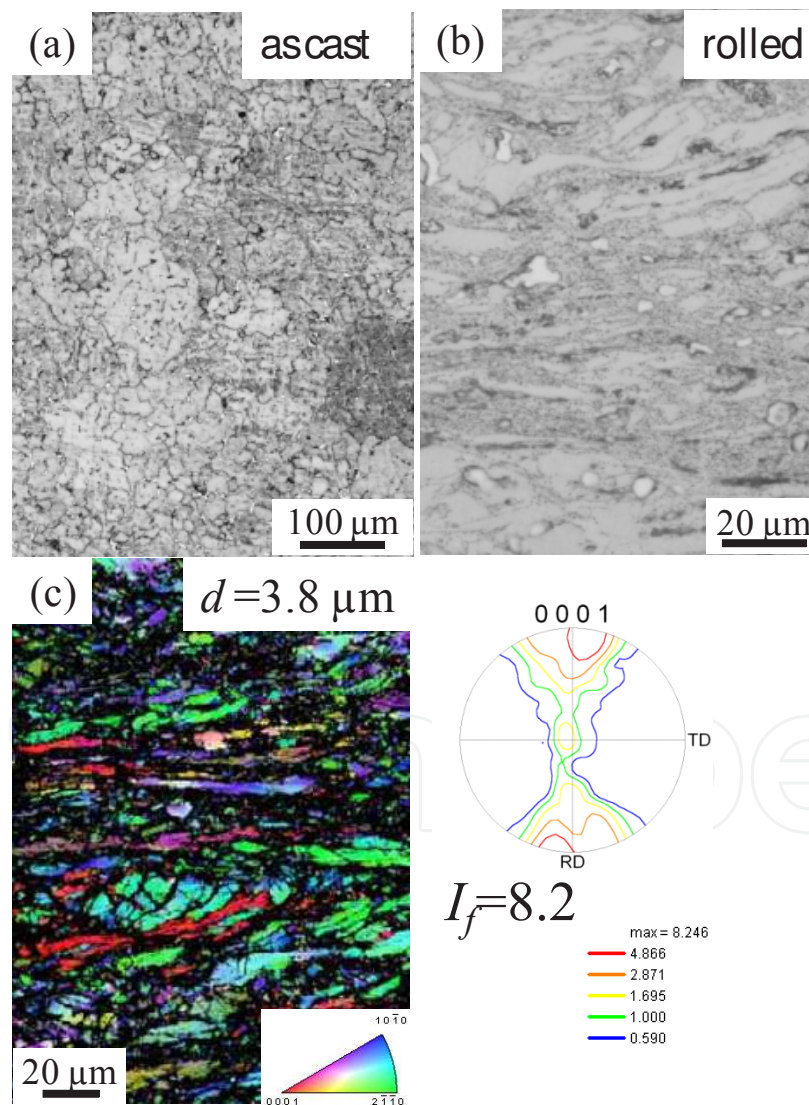

Figure 10. Optical micrographs (a)-(b) and inverse pole figure map (c) of as rolled AMX1001 alloy. The thickness of rolled materials was 1 mm (a total reduction in thickness of 75%).
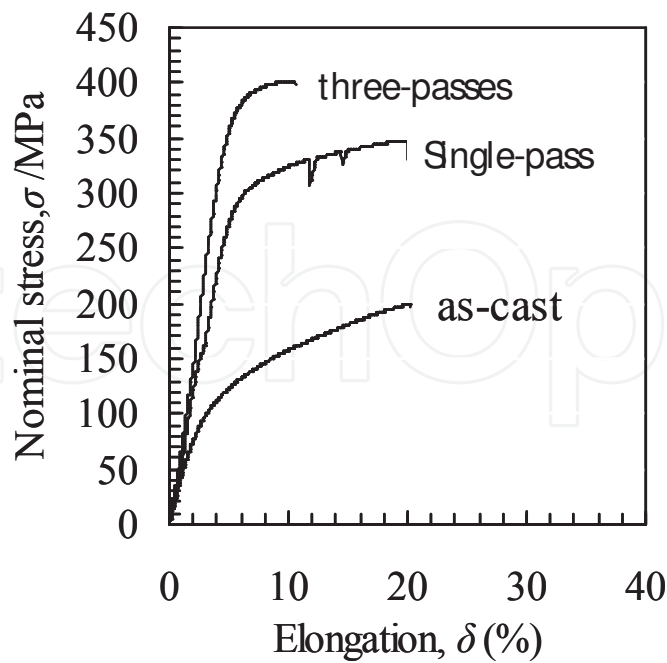
Figure 11. Nominal stress–strain curves for as-rolled AZX1001 alloy samples subjected to multipass rolling at 200 °C or single pass rolling at 400 °C.

3.4. Annealing conditions and the thermal stability of AMX1001 rolled sheet

Figure 12(a) shows the relationship between the mechanical properties and the annealing temperature for rolled sheets of AMX1001. Figure 12(b)-(e) shows optical micrographs of rolled sheets of AMX1001 annealed at various temperatures between 150 and 400 °C. The YS and UTS decreased by about 40 MPa and the elongation increased to 11% when the annealing temperature was raised from 150 °C to 200 °C. A further increase in the annealing temperature to 300 °C resulted in a significant decrease in the YS to 260 MPa and in the UTS to 310 MPa, whereas differences in the YS, UTS, and elongation for annealing temperatures between 300 and 400 °C were minimal. An AMX1001 rolled sheet subjected to annealing at 200 °C for 1 h showed no significant reduction in strength or tensile properties, which were are similar to those of high-strength Mg alloy. In other words, the AMX1001 alloy showed excellent thermal stability as a result of the addition of a small amount of Ca [10–11]. The addition of Al, however, did not appear to have any effect on the thermal stability. The changes in the strength and elongation of the AMX1001 rolled sheet suggest that static recrystallization occurs at 200 to 250 °C [Figures 12 (a) and (b)-(c)]. The deterioration in mechanical properties can be effectively controlled by suppressing grain growth. Figure 13 shows that Al–Ca compounds form along the grain boundaries after annealing, but that some compounds form in the grain. Focusing on the grain size, fine grains are seen in the material annealed at 300 °C, whereas 18-μm grains are seen in the material annealed at 350 °C; furthermore, there is also a decrease in the formation of Al–Ca compounds. As the annealing temperature was increased, the Al–Ca compounds were able to control the grain growth, and grain coarsening occurred rapidly at temperatures close to the solution temperature. The other elements in the alloy are considered to be partially

soluble at 400 °C, and the formation of Al_2Ca compounds has been reported to be effective in improving the ductility of alloys [13]. The alloy in this work, in which Al_2Ca compounds were formed as a result of the addition of 1 mass% of Ca, is considered to retain its ductility while showing a greater strength and larger elongation than other Mg alloys.
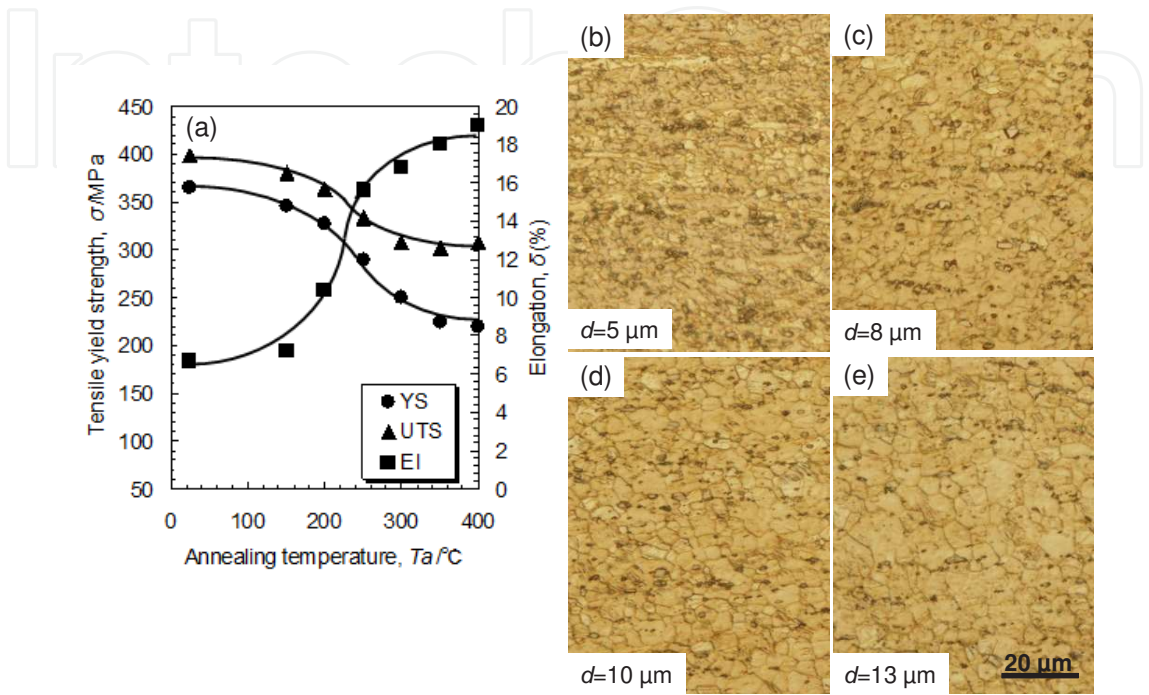

Figure 12. Tensile properties (a) and optical micrographs (b)-(e) of samples of AMX1001 alloys annealed at 200°C(b), 250°C(c), 300°C(d) and 400°C(e) for 1 h.
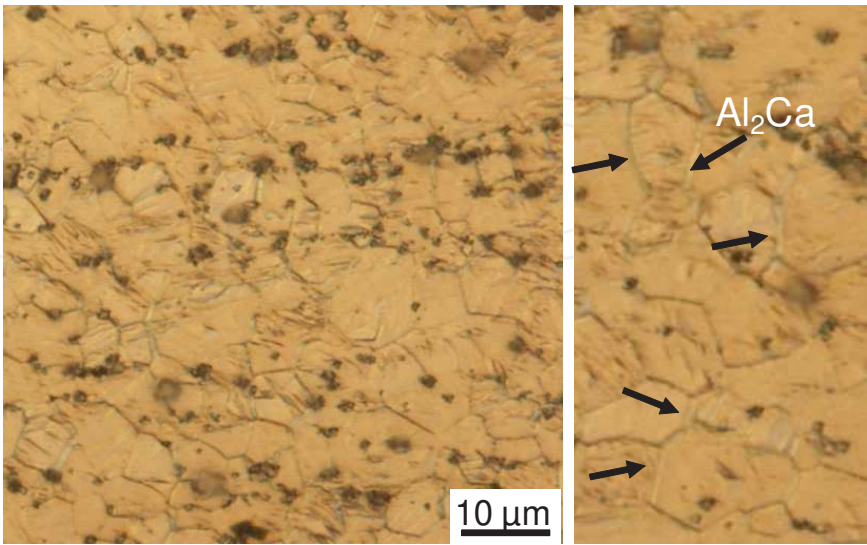

Figure 13. Optical micrographs of the typical dispersion state of Al–Ca compounds in the alloy after annealing at 400 °C for 1 h.
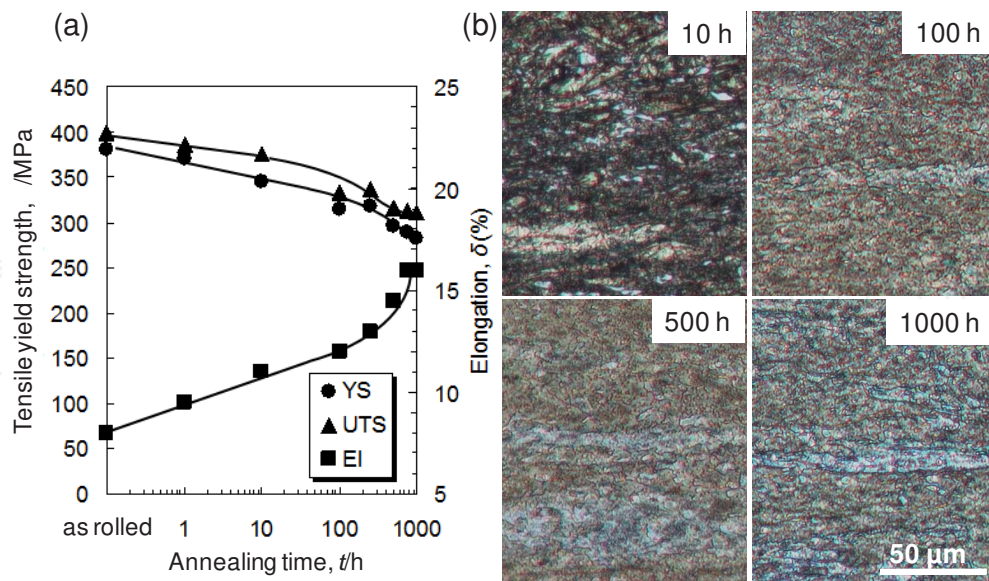
Figure 14. Relationship between the annealing time and the mechanical properties at room temperature of rolled samples of AMX1001 alloy annealed at 200 °C (a). Optical micrographs of rolled samples of AMX1001 alloy annealed at 200 °C for various holding times (b).

The rolled samples of AMX1001 alloy did not show any marked loss of strength or changes in microstructure on annealing at 200 °C for 1 h. We therefore extended the annealing time to 1000 h to test the thermal stability of the AMX1001 rolled sheet. Figure 14(a) shows the YS and elongation for a sample of rolled AMX1001 alloy annealed at 200 °C for various times up to 1000 h, as tested at room temperature. The YS and UTS decreased gradually with increasing annealing time, whereas the elongation markedly improved. The tensile properties of samples annealed at 200 °C for 1000 h did not depend on the annealing time. Figure 14(b) shows optical micrographs of samples annealed at 200 °C for various annealing time. Figure 14(a) shows that when the annealing temperature was maintained at 200 °C for 1000 h, even though the YS was reduced from 390 to 280 MPa, the elongation improved from 8 to 22%. Although the α-Mg phase grew from 4 to 10 μm, and a lamellar microstructure in which the Al_2Ca compounds was finely dispersed in the α-Mg phase was formed, no substantial changes in the microstructure were observed, even after annealing for 1000 h. An examination of the optical micrographs in Figure 14(b) shows that when the sample was annealed for 1000 h, the lamellar microstructures of the α-Mg and Al_2Ca compounds were the same as those observed before annealing. In other words, degradation of the mechanical properties of the AMX1001 rolled sheet after annealing at 200 °C is due to static recovery of the alloy. With regard to the reinforcing factor of this material, Al_2Ca compounds control the grain growth of α-Mg phase even after annealing at 200 °C for 1000 h. The formation of Al_2Ca compounds by adding 1 mass% of Ca is therefore an effective way of increasing the heat resistance of the alloy. The YS and UTS of rolled AMX1001 alloy were lower than those of Mg–Zn–Y extruded alloy [8] after heat treatment at 200 °C for 1000 h; however, AMX1001 alloy can be fabricated into thin rolled sheet at rolling temperature below 200 °C with a small number of passes. In the case of

Mg alloys, it is important that they retain a high strength and a high ductility if they are to be used as industrial materials, and the Ca-containing Mg alloy AMX1001 is a material that possesses such properties.

3.5. Formability and damping property of flame-resistant magnesium alloy

The formability of AMX1001 high-strength rolled sheets was examined by means of a conical cup tests performed at room temperature to 250 °C and an initial strain rate of 2.7×10^{-1} s^{-1}. Specimens measuring 36 mm in diameter were cut from AMX1001 rolled sheet and subjected to conical-cup tests, the results of which are shown in the Figure 15. For comparison, Figure 15 also shows the conical cup value for AZ61 Mg rolled sheet and for high-strength rolled sheet 6N01 Al alloy (Al–0.58Mg–0.6Si mass% alloy) [14], which has a YS of 480 MPa, a UTS of 497 MPa, and an elongation of 8%, and is used in high-speed rail vehicles. The conical cup value of AMX1001 rolled sheet was 27 at 150 °C or above; this value was not significantly improved by increasing the testing temperature to 200 °C. At test temperatures of up to 100 °C, the 6N01 Al rolled sheet showed a better formability than the AZ61 and AMX1001 Mg rolled sheet; however, at a test temperature of 150 °C, the conical cup values for the Mg and Al alloys were very similar. The conical cup value for the AMX1001 rolled sheet was therefore excellent at test temperatures of 150 °C or more. At high testing temperatures, samples of AZ61 and AMX1001 alloy produced by low-load processing showed higher conical cup values than did high-strength 6N01 Al rolled sheet. AMX1001 rolled sheet fabricated by the rolling process described in this study therefore showed better formability than fine-grained 6N01 Al rolled sheet. Figure 16 shows optical micrographs and IPF maps of AMX1001 rolled sheet after conical cup testing at 150 °C and 200 °C. The observation area was 500 μm from the fracture tips of the crown part. The microstructure after conical cup testing at 150 °C showed an elongated grain in comparison with the as-rolled microstructure shown in Figure 10. Along with the improvement in formability demonstrated by the conical-cup test, equiaxial grains were formed in the microstructure at a testing temperature of 200 °C; additionally, the nonrecrystallized region changed to a recrystallized region and a fine grain structure formed with without elongation when the temperature of deformation was high. In other words, to improve the formability of the Mg alloy, it is necessary to select an appropriate temperature and to make use of DRX during plastic deformation. The IPF maps shown in Figure 16 show that at a testing temperature of 200 °C, crystal orientation was random and the texture of the sample after the conical-cup test was weak in comparison with that observed after testing at 150 °C. We found that at a testing temperature of 200 °C, DRX occurred during plastic deformation.

It is well known that Mg alloys have excellent vibration-absorbing properties. The vibration properties of Mg alloys are often reported [17–18], but few comparisons have been made with steel or Al alloys. We examined the damping properties of samples of rolled and/or extruded steel, Al alloys, and Mg alloys by analyzing the waveforms produced after a displacement of 0.45 mm by performing a cantilever vibration test. The cantilever test specimens measured 18 mm wide, 200 mm long, and 1 mm thick. We investigated the damping ratio at a strain amplitude of 2×10^{-4} s^{-1} and a displacement of 0.45 mm at the tip of the cantilever. The damping properties of steel (SUS 304), aluminum alloys (A7075, A5083, A6063, A6N01), and magnesium

Figure 15. Appearance of conical cup sample and the relationship between the conical cup value and the testing temperature. The conical cup test was performed at various temperatures at an initial strain rate of 2.7×10^{-1} s^{-1}.

Figure 16. Optical micrographs (a), (b) and inverse pole figure maps (c), (d) of AMX 1001 rolled sheet after conical cup testing at 150 °C (a), (c) and 200 °C (b), (d) at an initial strain rate of 2.7×10^{-1} s^{-1}.

alloys (AZ series, Mg-RE, AZX, and AMX) are shown in Figure 17. From Figure 17, the damping properties improved in the order Mg alloys ≥ Al alloys ≥ steel. The damping ratios of the Mg alloys were dependent on the type of alloy, as in the case of the mechanical properties, and they were affected by the nature of the elements forming the alloy. However, the variation in

the percentage of damping ratio on changing of elemental content was small. The material type and the damping ratio showed a linear relationship. In the case of Mg alloys, alloys in the region between the AZ series of Mg alloys and Mg–RE alloys, where flame-resistant Mg alloys occur, had damping properties that were inferior to those of commercial AZ-series Mg alloys, showing that these properties are weakened by the addition of Ca. However, no effect of the addition of Al on the damping ratio could be identified.

Figure 17. Damping properties of various rolled or extruded materials.

4. Summary

We investigated various properties of flame-resistant Mg alloys. By subjecting TRC materials to a total reduction in thickness of up to 75% by multipass rolling without reheating, we produced a rolled sheet with a tensile strength of 400 MPa and an elongation of 8%. During the multipass rolling process, grain refinement occurred as a result of dynamic recrystallization of the Mg phase and crushing of Al_2Ca compounds. A study of the heat-treatment properties of AMX1001 high-strength rolled sheet found that the yield strength was reduced from 330 to 250 MPa on heating at between 200 and 300 °C for 1 h, whereas the elongation improved from 8 to 17%, suggesting that by static recrystallization had occurred. When we investigated the mechanical properties after heating the material at 200 °C for 1000 h, the tensile strength reached 280 MPa and the elongation improved to 22%. In other words, AMX1001 rolled sheet have an excellent thermal stability. Furthermore, the conical cup value for AMX1001 rolled sheet was maximal at a test temperature of 150 °C or more, and this value was superior to that

of fine-grained rolled A6N01 alloy. A study of the damping properties of various alloys showed that they improved in the order steel ≤ aluminum alloys ≤ magnesium alloys. Overall, the properties of high-strength AMX1001 rolled sheet are superior to those of fine-grained aluminum alloys. In particular, this Mg alloy shows excellent thermal stability, damping properties and formability.

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