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Influence of the processing route on the mechanical properties at high temperatures of Mg-Ni-Y-RE alloys containing LPSO-phases

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Abstract

The mechanical properties at high temperatures of two Mg-Ni-Y-RE alloys prepared by conventional or powder metallurgy routes have been evaluated. All alloys exhibit high-strength up to 250°C but their strength depends more on the composition of the alloy rather than on the processing route. High strength of the alloys arises from their fine grain size but there is an additional contribution of load transfer from the magnesium matrix towards the second phases in the alloys processed by the conventional route while additional hardening in the alloys prepared by powder metallurgy is due to Orowan mechanism. Above 250°C, the strength drops to very low values but all alloys exhibit high elongations and grain boundary sliding is the controlling mechanism. Grain boundary sliding also induces cracking of the coarse second phases into smaller pieces during the initial stages of plastic deformation of conventionally processed alloys.

Keywords: A. Mechanical characterization; B. Magnesium alloys; B. LPSO-phase, C. Processing route; D. Deformation mechanisms.

1. Introduction

The mechanical properties of magnesium alloys can be substantially improved by the addition of proper alloying elements [1-4]. Simultaneous addition of transition metal (TM) and rare earth (RE) elements has been proved to harden considerably magnesium, attaining the maximum strengthening when the alloys are reinforced by long period stacking ordered structures (usually

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named as LPSO-phases) [5-16]. Most of the work has been focused in Mg-Zn-(Y,RE) alloys [10-20], although new Mg-Ni-(Y,RE) alloys have been developed more recently [7,21-24]. Difference among Ni-containing and Zn-containing alloys results from nickel insolubility in the magnesium lattice while zinc exhibits certain solubility. Nickel excess appears in Mg-Ni-Y alloys containing the LPSO-phase as Mg_2Ni [21,22,25] while zinc can form binary and ternary intermetallic Mg-Zn compounds and/or remain in solid solution within the magnesium lattice [16,18,26,27].

The addition of insoluble rare earth elements such as lanthanum or cerium to ternary alloys results in formation of intermetallic Mg-RE compounds [14,16,17,23]. Due to high atomic Mg/RE ratio in intermetallic Mg-RE phases, small additions of RE elements leads to significant volume fractions of these phases in Mg-Ni-Y-RE alloys [23]. Hardening effect of Mg-RE phases has been reported to be smaller than that associated with LPSO-phases [16,18]. Major effect of Mg-RE phases, however, is to increase the ability of the alloy to deform superplastically [21].

Although the microstructure of Mg-Ni-Y-CeMM alloys processed by a conventional route of extrusion of as-cast rods is fine (the grain size oscillates between 2 and 5 μm), additional refinement could be achieved by using powder metallurgy techniques. The processing route determines the mechanism controlling the deformation at room temperatures, which is associated with the size and distribution of second phases [28]. This effect could be also expected at higher temperatures. Thus, the aim of this research has been to identify the mechanisms controlling the deformation in the temperature range 100-400°C of two Mg-Ni-Y-CeMM alloys prepared through two different processing routes.

2. Material and Methods

Two alloys of nominal compositions, given in atomic percentage, $Mg_{96}Ni_2Y_1CeMM_1$ (designated as MgNi2 alloy) and $Mg_{94}Ni_3Y_1CeMM_1$ (designated as MgNi3) were obtained from eutectic binary Mg-Y, Mg-RE and Mg-Ni master alloys, adjusting the composition with minor amounts of the pure elements (Mg, Ni, Y) and Ce-rich mischmetal (53 wt. % Ce, 26 wt. % La, 16 wt. % Nd, 5 wt. % Pr). The alloys were casted in bars of 42 mm in diameter. Some of the bars were machined up to 40 mm and hot extruded at 400°C using an

extrusion ratio of 18:1. These materials will be designated as CP (conventional processing)

The remaining bars were remelted to produce rapidly solidified powders (<100 μm) through the Electrode Induction-Melting Gas Atomization technique. The powders were isostatically cold pressed and then extruded at 400ºC using an extrusion ratio of 18:1. These materials will be designated as PM (powder metallurgy)

The microstructure of CP and PM alloys was characterized by optical microscopy (OM) and scanning and transmission electron microscopy (SEM and TEM, respectively), while the identification of the phases was performed by X-ray diffraction (XRD) and EDX microanalysis. A complete microstructural characterization of the alloys can be found elsewhere in reference 24.

Tensile mechanical properties were evaluated in the temperature range 100-400°C. Cylindrical samples of 3 mm radius and 10 mm gauge length were machined from the extruded bar with their longer dimension parallel to the extrusion direction. Tensile tests were performed at an initial strain rate of 10^{-4} s^{-1} in a universal Instron tensile machine. The stress exponent was determined through jump strain rate tests. In these tests, the specimens were initially deformed at a strain rate of 10^{-4} s^{-1} until a steady state was reached. The strain rate was then reduced to 10^{-5} s^{-1} until a new steady state was again attained. Successive changes in the strain rate were carried out following this procedure.

3. Results

The microstructure of the extruded alloys is presented in Fig. 1. The microstructure consists of a fine-grained magnesium matrix embedding a high volume fraction of second phases. Second phases are coarse and arranged in strings along the extrusion direction in CP alloys while they are equiaxed and much finer in PM alloys. The grain size was fine for all alloys, especially for both PM alloys. It was 4 μm for CP-MgNi2, 2 μm for CP-MgNi3 and 0.5 μm for the PM alloys. The volume fraction of second phases was 27 and 52 % for the MgNi2 and MgNi3 alloys, respectively. To get a more complete description of the microstructure of all alloys, the readers are referred to a previous work [28]

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True-stress-true strain curves between 100 and 400°C at 10^{-4} s^{-1} are presented in Fig. 2 for all materials while the evolutions of yield stress, maximum strength and elongation to failure with the test temperature are presented in Fig. 3. According to the data, two different behaviours can be clearly distinguished depending on the temperature, with a transition at about 250°C: (a) At low temperatures, below 250°C, all the alloys exhibit high yield stresses. (b) Above 250°C, however, the yield stress drops to very low values. This significant decrease in strength is accompanied by a considerable increase in the elongation to failure, especially in the case of CP alloys.

3.1 Low temperature interval (100-200°C)

At 100 and 200°C, the maximum strength always corresponds to the high-alloyed material (MgNi3 alloy), although the differences tend to decrease gradually with increasing the test temperature. Furthermore, for a given composition, the alloy processed by powder metallurgy always attains the maximum strength. At 100°C, the yield stress oscillates between the 402 MPa for the PM-MgNi3 alloy and the 220 MPa for the CP-MgNi2 alloy. The ductility of both MgNi3 alloys is very low, not higher than 2 %. On the contrary, CP-MgNi2 and PM-MgNi2 combine high strength with some ductility. Furthermore, elongation to failure of the PM-MgNi2 alloy was almost twice that of CP-MgNi2 alloy (**21** % against **11** %). The different ductility among CP-MgNi2 and PM MgNi2 alloys, also found at room temperature [28], results from the different deformation mechanism operating in each alloy. The fine equiaxed microstructure of PM MgNi2 alloy promotes the stress is uniformly supported by all the grains. Stress distribution is more heterogeneous in the case of CP2-MgNi2 alloy because magnesium grains transfer load into the coarse second phases. When second phase cracks, the load comes back to the magnesium grain and, if the stress is not supported by the magnesium grains or by other second phases, nucleation and propagation of the crack proceeds catastrophically. As this behaviour was also reported at room temperature [28], it can be concluded that the use of the powder metallurgy route induces not only a benefit on the strength but also on the ductility of the MgNi2 alloy while the second phases can harden the PM alloy.

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At 200°C, the highest yield stress is found for the PM-MgNi3 (270 MPa), but the maximum strength corresponds to the CP-MgNi3 (335 MPa) because of its greater hardening during plastic regime. Precisely, the absence of hardening in PM alloys during plastic deformation at 200°C is the most noticeable difference with CP alloys. Unlike at 100°C, the ductility is practically the same for CP-MgNi2 and PM MgNi2 alloys. Continuous load transfer from the magnesium matrix induces continuous hardening during plastic flow (see tensile curves of Fig. 2b) which promotes large elongations than that found at 100°C. On the other hand, the fine precipitates of the PM-MgNi2 alloy are not effective obstacles for dislocation motion, favouring premature failing of the material with elongations around 21 %, very close to the value found at 100°C.

It is interesting to note the presence in the true stress-true strain curves of a yielding phenomenon restricted exclusively to the MgNi2 alloy at 100 and 200°C. At 100°C, the stress for the PM-MgNi2 alloy remains almost stable up to 3 % of strain and, then, the hardening stage commences. In the case of the CP-MgNi2 alloy, the yield point is followed by a slight decrease in the stress, immediately followed by the hardening stage. At 200°C, both MgNi2 alloys exhibit also the yield point phenomenon but hardening takes place in CP-MgNi2 alloy while a steady state is attained in the PM-MgNi2 alloy once the upper yield point is surpassed. The occurrence of yield point phenomenon in magnesium alloys is usually associated with the segregation of tiny particles within the magnesium grains [29-31]. These particles pin down or anchor dislocations, causing an increase in the stress up to a critical value is attained (upper yield stress). Once the upper yield point is reached, there is a load drop due to unpinning of dislocations which is more noticeable in the PM alloy because of the higher volume fraction of fine precipitates inside the magnesium grains. Probably, the absence of yield point phenomenon in tensile curves of MgNi3 alloys is due to the low volume fraction of the magnesium phase (only 48 %) which can dilute or mask the occurrence of such process in tensile curves.

3.2 Transition temperature (250°C)

Transition from the high-strength behaviour at low temperatures to that of low strength at high temperatures is noticed at 250°C. Compared to 200°C, the yield stress experiences a significant decrease although it still remains relatively high.

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Nevertheless, the degree of the decrease is minimised in the case of conventionally processed alloys. Thus, for instance, the yield stress decreases about 60 % for PM-MgNi3 alloy (from 272 MPa at 200°C to 111 MPa at 250°C) against the 32 % for the CP-MgNi3 alloy (from 256 MPa at 200°C to 175 MPa at 250°C). Elongation to failure of PM alloys, about 100-125%, was almost twice that of CP alloys.

3.3 High temperature interval (300-400°C)

The yield stress drops off below 60 MPa independently of the composition of the alloys and the processing route. These values decrease below 5 MPa when the temperature rises up to 400°C (see Fig. 3). All curves show certain hardening in the course of plastic deformation as temperature increases, being more significant for the CP-MgNi2 alloy. Such hardening arises, probably, from grain coarsening during plastic deformation and it results in a considerable increase in stress. Usually, the maximum stress can be 1.5-3 times higher than the stress in the yield stress.

3.4 Calculation of stress exponent

Fig. 4 plots the strain rate-true stress in the steady state in a double logarithmic at different temperatures for all materials between 100 and 400°C according to the general-power law constitutive creep equation

$$\dot{\varepsilon} = K\sigma^n e^{(-Q/RT)}$$

where K is the creep constant, n the apparent stress exponent, σ the stress in the steady state, Q the apparent activation energy, T the absolute temperature and R the universal gas constant. Table 1 lists the values of the stress exponent *n* at different temperatures for all the alloys. At 100°C only data of MgNi2 alloys are presented because of the low ductility of MgNi3 alloys. Although a complete steady state is not attained in tensile curves of the CP-MgNi2 alloy, strain rate jumps were carried out in very short strain intervals (2.5 %) in which no relevant microstructural changes are expected, as confirmed by the excellent correlation among the different points. The stress exponent *n* takes very high values; 22 and 35 for CP-MgNi2 and PM-MgNi2 alloy, respectively. At 200°C, the highest n

corresponds to CP-MgNi2 alloy, n=46, being 29 for the PM-MgNi2 alloy. It is interesting to remark that n for both MgNi3 alloys is significantly lower than for MgNi2 alloys (16 for CP-MgNi3 and 18 for PM-MgNi3). At 300°C, the value of n depends on the strain rate. As a general rule, n takes values around 5-8 at high strain rates, corresponding the lowest values to PM alloys and the highest values to CP alloys. At low strain rates, however, n is close to 2-3. In addition, new transition towards values higher than 5-6 is observed for the PM alloys at the lowest strain rates. As the temperature increases, all the intervals with different n are shifted to higher strain rates, as can be checked in Table 2. Moreover, the region in which n is close to 2 seems to be wider as the temperature increases. For instance, n is about 2.5 in the 10^{-5}-10^{-2} s^{-1} interval at 350°C for CP-MgNi3 material.

The activation energy of the process was determined in the superplastic regime, n close to 2-2.5, plotting in a double logarithmic manner the σ/E against the inverse of the absolute temperature. For CP alloys, the activation energy is 181 kJ/mol (at stress of 20 MPa) and 214 kJ/mol (at stress of 30 MPa) for CP-MgNi2 and CP-MgNi3 alloys, respectively. In the case of PM materials, the values are 208 kJ/mol (at stress of 44 MPa) and 294 kJ/mol (at stress of 38 MPa) for PM-MgNi2 and PM-MgNi3 alloys, respectively. These energies are much higher than the activation energy for grain boundary (92 kJ/mol) or lattice diffusion (135 kJ/mol) in magnesium [32,33], but they are not so far from the activation energies for LPSO-containing magnesium alloys which range between 182 and 276 kJ/mol [17,34-36]. This suggests that activation energy for alloys containing high volume fractions of LPSO-phases is higher than for diluted magnesium alloys. This indicates that interfaces or LPSO-phase itself could play a significant role during the superplastic regime of the alloys.

3.5 Microstructure of deformed samples

Microstructural examination of the deformed samples has been carried out in the three intervals in which the mechanical behaviour has been divided. The observations evidence differences in the mechanism controlling plastic deformation of CP and PM alloys, independently of the composition of the alloy. This clearly confirms that the processing route determines markedly the deformation behaviour of Mg-Ni-Y-CeMM alloys rather than their composition.

Fig. 5 shows the microstructure in two regions of a CP-MgNi2 sample tested at 200°C at 10^{-4} s^{-1}. The microstructure in Fig. 5a resembles that of the non-deformed alloy and corresponds to the region outside the necked region. On the other hand, Fig. 5b shows the microstructure in the necked area. Here, two main changes occur in the microstructure of the alloy: Firstly, second-phase particles, especially $Mg_{17}RE_2$ particles, are fragmented. Secondly, magnesium grains are elongated along the tensile direction. In the case of PM alloys, some cavities were developed in the course of the deformation in the region close to the fracture, as shown in Fig. 6a. Most of second phase particles remain intact outside the necked region (see Fig. 6b) but the number of cracked particles increases progressively towards the fracture surface, within the necked region, as shown in Fig. 6c. Etched samples demonstrate that initially equiaxed grains are elongated along the tensile direction, especially those grains close to the fracture region, as shown in Fig. 7a-c.

At the transient temperature of 250°C, no significant changes are noticed with respect to samples deformed at 200°C. Major difference is that cracking of $Mg_{17}RE_2$ particles occurs from the earliest stages of plastic deformation over the entire gauge length, although the amount of fragmented particles is lessened outside the necked area (see Fig. 8). Etched samples reveal magnesium grains severely deformed along the tensile direction within the necked region, especially those grains close to the fracture surface (see insets of Fig 8). In the case of PM alloys, small cavities were observed, mainly concentrated in the region close to the fracture. The tendency for cavity development is higher in the PM-MgNi3 alloy, as can be seen in Fig. 9. Etched samples proved that magnesium grains are deformed for the PM-MgNi2 alloy and hardly deformed for the PM-MgNi3 alloy (Fig. 10).

Microstructural evolution during the tensile test is completely different for the alloys deformed above 250 °C. In the case of CP alloys, considerable refinement of second phases takes place from the initial stages of deformation, as shown in Fig. 11 for the CP-MgNi3 alloy. Moreover, the small fragments resulting from the cracking of second phases are redistributed homogeneously throughout the magnesium matrix in such a way that the arrangements of second phases, initially existing as strings perfectly orientated along the extrusion direction, disappear in the course of plastic deformation (see Fig. 12).

In addition, magnesium grains retain their equiaxed shape and only at the highest test temperature of 400°C some grain coarsening is noticed, although grain growth is hampered in the deformed region when compared with grain size in the non-deformed heads of tensile samples. At 300°C some cavities are found in the deformed region, but the tendency to form cavities decreases with increasing the test temperature. For instance, no cavities are found in the sample deformed 250 % at 400°C. Another significant change in the microstructure is recrystallization of coarse-grained areas during straining. At 300°C recrystallization is partial but it is total at 400°C (see Fig. 12). In the case of PM alloys, the most noticeable feature found in samples tested at temperatures higher than 250°C is the high propensity to cavitation, especially for the PM-MgNi2 alloy, as observed in Fig. 13. Cavities are usually concentrated in the necked region, especially close to the fracture surface. They are arranged as long strings, up to 30 μm in length and 2 μm in width, aligned along the tensile direction. Occasionally, these strings can coalesce, originating cavities up to 20 μm width. The size and number of cavities decrease progressively as moving away from the fracture surface, adopting the appearance of fine porosity far away from the fracture region.

4. Discussion

The results of this study reveal that high-strength of Mg-Ni-Y-CeMM alloys can be retained up to 250-350 MPa at 200°C and around 175-250 MPa at 250°C. These values are much higher than those reported for commercial magnesium alloys such alloys belonging to AZ, AM, AS, ZK or ZE families in which the strength falls down to very low values when the temperature exceed 100-150°C [3]. Furthermore, the maximum strength is higher than those of commercial alloys such as WE43 or WE54 which are designed for applications up to 200°C, with the advantage that the maximum strength is achieved without long ageing treatments. The same behaviour is found in the same alloys when they are processed by extrusion of rapidly solidified ribbons [37]. In this case, the maximum strength can attain about 500 MPa at room temperature, although these values decrease rapidly above 100-150°C, even in the case of WE43 or WE54 alloys.

Tensile tests evidence how the composition and the processing route determine the mechanical strength of Mg-Ni-Y-CeMM alloys between 100 and 400°C. The intervals agree rather well with the two different behaviours identified through tensile testing. At temperatures up to 200°C, the strength depends more on the composition of the alloy than in the processing route, as found at room temperature [28]. The strength of MgNi3 alloys is higher than for MgNi2 alloys because of the higher volume fraction of hard second phases. The effect of alloy composition in the mechanical properties, however, becomes irrelevant beyond 200°C because above this temperature the higher strengths correspond to CP alloys.

The microstructure among the alloys prepared following the same processing route is very similar, varying only the volume fraction of second phases (about 27 % for the MgNi2 alloy and about 52 % for the MgNi3 [28]). Major differences between CP and PM alloys are:

(1) Type of the second phases. Common phases for the alloys processed by both routes are Mg, intermetallic Mg-RE phases, Mg_2Ni and LPSO-phase while fine $Mg_{24}Y_5$ precipitates are exclusively present in both PM alloys.

(2) Shape, size and morphology of the phases. Second phases in PM alloys are finer and more equiaxed than in CP alloys.

(3) Their distribution in the magnesium matrix. Second phases are homogeneously distributed in the magnesium matrix in PM alloys while they are concentrated in bands as strings of coarse particles arranged along the extrusion direction in the case of CP alloys.

(4) Grain size of the magnesium matrix. Around 0.5 μm for PM alloys and smaller than 4 μm for CP alloys (2 and 4 μm for CP-MgNi3 and CP-Mg-Ni2 alloys, respectively). It is interesting to note that there are coarse non-recrystallised areas in CP alloys, especially in the case of the CP-MgNi3 alloy.

(5) Size and morphology of the LPSO-phase. LPSO-phase appears as long blocks, up to 20-30 μm in length, oriented in the extrusion direction in CP alloys while it appears as long lamella up to 500 nm length and 1 nm thick within the magnesium grains in PM alloys.

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These microstructural differences should account for the different mechanical behaviour found between CP and PM alloys.

At 100°C and 200°C, the maximum strength corresponds to MgNi3 alloys, as also found at room temperature [28]. The main strengthening effect is related to the fine grain size of the alloys and the higher volume fraction of second-phase particles, 52 % in MgNi3 alloys compared to 27 % in MgNi2 alloys. For CP alloys hardening is mainly attributed to high contribution of Hall-Petch mechanism due to the fine grain size of the alloys but load transfer from the magnesium matrix to the coarse second phases also has a significant contribution because Young moduli of Mg-RE compounds [39] and LPSO phase [39-41] is higher than that of the magnesium matrix. For PM alloys, contribution of grain boundary strengthening is still higher because of the fine-grained magnesium matrix (around 0.5 μm) although the reinforcing due to the multiple phases existing in PM alloys can be also relevant through other two mechanisms: (i). Composite strengthening, due to load transfer from the magnesium matrix towards second-phase particles of about 0.5 μm in size homogeneously dispersed in the magnesium matrix, is manifested by the presence of isolated second phase particles broken in the deformed samples, as seen in Fig. 6c. (ii) Hardening induced through Orowan mechanism by dislocations interacting with thin LPSO-phase plates and fine $Mg_{24}Y_5$ precipitates inside the magnesium grains.

All alloys show high n values at 100 and 200°C, always above 15, indicative that the deformation is controlled by dislocation motion. Such high values are generally attributed to the existence of a threshold stress. It is interesting to note that n values are much higher than those reported for PM magnesium alloys [42-45] or magnesium composites [46-48], with a maximum value of 19 at 200°C for strongly textured PM Mg [44]. This indicates that high n values in Mg-Ni-Y-CeMM alloys are associated with the second phases present in the alloy rather than with the fine oxide dispersion existing in PM alloys [43]. Moreover, n depends not only on the processing route but also on the temperature and alloy composition. At 100°C n is much higher for PM-MgNi2 alloy than for the CP MgNi2 alloy, 35 against 22. This indicates that dislocation motion is more hindered in the PM alloy than in the CP alloy. This difference in the n value should result from large contribution of a mechanism operating in the PM alloy

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that hardly contributes in the CP alloy. This mechanism should correspond to hardening induced by the fine second phases inside the magnesium grains (Orowan strenghtening). Orowan hardening is significant up to 100°C because the distance between precipitates inside the magnesium grains is small [28], resulting in a strain hardening during the plastic flow of the PM-MgNi2 alloy comparable to that found in the CP-MgNi2 alloy. At 200°C, however, Orowan hardening becomes less effective because dislocations can overpass more easily these obstacles by temperature assisted mechanisms such as dislocation glide or dislocation climb, so this mechanism does not induce hardening during the plastic flow. Nevertheless, the contribution of load transfer mechanism becomes higher, as noticed from a higher amount of broken second-phase particles and the constant strain hardening observed in the course of plastic deformation at 200°C. The subsequent balance results in a decrease in n from 35 at 100 °C to 29 at 200°C. Such effect is also reflected in the strain hardening of the samples tested at 100 and 200°C. Similar behaviour is found for the PM-MgNi3 alloy, although the material tested at 200°C still shows a slight hardening during plastic deformation due to the smaller volume fraction of the magnesium phase reduces the contribution of Orowan and load transfer mechanisms to the total strength of the alloy compared to that in the PM-MgNi2 alloy. This accounts for the lower n calculated for the PM-MgNi3 alloy compared to the PM-MgNi2 alloy, 16 and 29 respectively at 200°C.

On the other hand, n for the CP-MgNi2 alloy shifts from 22 at 100°C to 46 at 200°C. That is, the strain rate sensitivity of the alloy is greatly decreased as the temperature is increased within this temperature interval. This implies that dislocation motion is significantly hindered at 200°C. Such high n values can be explained considering load transfer from magnesium grains to coarse second phases (almost equiaxed $Mg_{17}RE_2$ and elongated LPSO-phases blocks) as the main mechanism controlling plastic deformation. If the alloys are considered as a composite, the ratio of the strain rates of the composite and the metallic matrix is given by $(1-\alpha)^n$, where α is the load-transfer coefficient ranging from 0 (no load transfer) to 1 (full load transfer) and n is the stress exponent [48-52]. Apparently, not significant changes in n values could be expected if the load transfer mechanism is the same at low temperatures. Nevertheless, the mechanical behaviour of the phases evolves in different ways with increasing

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the temperature; (i) Magnesium softening due to critical resolver shear stress of main slip systems in magnesium drops to very low values above 150°C [53]. Consequently, the stress transferred by the magnesium grains towards the second phases becomes proportionally higher as the test temperature increases, (ii) The high strength of the LPSO-phase is retained but its ductility increases gradually up to 200°C [54,55] and (iii) hardness of Mg-RE compound is the highest among the phases existing in the alloy [16,18] but its ordered structure renders this phase inherently brittle. Major brittleness of the Mg-RE phase has been reported for Mg-Zn-Y-RE alloys with different Y/RE ratios [18]. The strength and ductility of these alloys were improved as increases the Y/RE ratio, i.e. as the volume fraction of the LPSO-phase prevails over that of Mg-RE phase. At low temperatures, both Mg-RE and LPSO-phase are brittle but the fine-grained magnesium matrix is also very hard in such a way that the failure takes place when the additional stress supported by the second phases is not much higher than the applied stress. This is confirmed by the absence of cracking in any kind of particle outside the fracture surface [28]. On the other hand, observation of deformed samples only reveals multiple cracking of Mg-RE particles, even out of necking region but not of the LPSO-phase. This means that intermetallic Mg-RE particles cannot bear the load transferred by the magnesium matrix. Moreover, the stress released when these particles are broken must be re-transferred towards the LPSO-phase because this phase combines high yield strength as well as high ductility and considerable strain hardening in the course of plastic deformation within this temperature interval [54,55]. This different behaviour of Mg-RE and LPSO-phase particles with the temperature changes the load transfer contribution of each phase. The stress transferred below 200°C is low and it can be assumed by both Mg-RE and LPSO-phase particles until cracking of these phases takes place, resulting in lower n values. At high temperatures, magnesium matrix may transfer a significant part of the stress which is mainly supported by the LPSO-phase, leading to high n values.

It is interesting to note that n at 200°C is much higher for the CP-MgNi2 alloy than for the CP-MgNi3 alloy (46 against 18), so load transfer should be more effective in the former alloy. The lower volume fraction of magnesium phase in the CP-MgNi3 alloy implies a reduction in the load transfer contribution to

strengthen the alloy. Furthermore, second-phase particles not in direct contact with magnesium grains will not aid in load transfer. This means that load transfer in CP-MgNi2 alloy could be enlarged due to a more homogeneous distribution of the second phases which maximizes the number of magnesium grains transferring stress to second-phase particles.

Above 250°C, the strength of all alloys falls down to very low values. In addition, n values decrease up to 2-8 depending on the material, temperature and strain rate interval. At all temperatures, n values around 2-3 can be found within a certain strain rate range in which the alloys exhibit high elongations to failure, (up to 720 % for the CP-MgNi2 alloy deformed at 350°C at 10^{-4} s^{-1}, as shown in Fig. 3c). The relatively equiaxed shape of magnesium grains after the deformation, the low stress at which plastic flow progresses and n values close to 2, suggest the deformation under these conditions is controlled by grain boundary sliding (GBS). Nevertheless, the mechanism proceeds in different ways in CP and PM alloys. The deformation mechanism in CP alloys is very complex due to concomitant mechanisms can operate in the course of plastic deformation [17,56]. The initial microstructure evolves continuously during the plastic deformation. The sequence of microstructural changes is the following [56]: (1) Grain boundary/interface sliding from the initial stages of plastic deformation. The size of second-phase particles is too big to expect mutual sliding during the initial stages of deformation. Nevertheless, a good interface, free of defects, permits the sliding of magnesium grains along the long interfaces with the second phases existing in the alloy, i.e. Mg-RE/Mg and LPSO-phase/Mg interfaces, without cavity formation. The role of interfaces in the deformation mechanism is manifested by the high activation energy calculated, higher for the alloy containing a higher volume fraction of second-phases. (2) Cracking of second phase particles. To accommodate sliding without cavity formation, dislocations must be generated at both sides of the sliding interfaces. At the strain rate used for tensile tests, 10^{-4} s^{-1}, such dislocations can be easily removed from the interface in the magnesium phase but not in the case of the Mg-RE compounds and LPSO-phase because of their ordered structures. As a consequence, sliding induces dislocations pile-ups in the second-phase particles at the vicinity of the sliding interface, which can result in considerable local stresses accumulation [17]. If a critical stress is

exceeded, cracking of the second-phase particles takes place, as experimentally observed. This mechanism explain LPSO-phase cracking after very short strains at very low applied stresses while such cracking is absent at 250°C at much higher applied stresses. (3) Homogeneous redistribution of second phases in the magnesium matrix. Microstructural observations clearly demonstrate that fragments resulting from multiple fractures of second phases are redistributed into the magnesium matrix. Such redistribution implies certain modification in the mechanism controlling the superplastic flow with respect that occurring during the initial stages. Refinement of large particles into smaller ones would permit the rotation of magnesium grains during grain boundary sliding [57,58]. This mechanism should permit the gradual displacement of second-phase pieces inside the magnesium bands, initially free of them. Cracking of the second phases predominates during the initial stages and becomes less important as the deformation proceeds. This accounts for the limited particle redistribution found after high elongations, as shown in Fig. 14. The microstructure of the samples deformed at 300°C up to 110 % resembles rather well the initial microstructure, although the refinement of second phases is noteworthy. On the contrary, grain rotation is favoured as the size of second-phase fragments is reduced. Once this mechanism becomes predominant, the distribution of the small second-phase particles occurs quickly, as deduced from comparison of the sample deformed 120 and 250 %. (see Fig. 14). (4) Recrystallization of non-recrystallised regions. It is well known that a fine-grained structure is a requisite for the occurrence of GBS. Non-recrystallised areas could constitute an obstacle for GBS/grain rotation.

The extremely large elongations of CP-MgNi2, almost two times that of CP-MgNi3 alloy, is due to the optimal volume fraction of second phases. The volume fraction of second phases is 27 % for CP-MgNi2 and 52 % for CP-MgNi3. Microstructural observations reveal the excellent compatibility among the different interfaces for sliding during the initial stages of plastic deformation, leading to fracture of second phases. Then, grain rotation of magnesium grain is accommodated by the cracked second phases, resulting in the gradual redistribution of second phases. Once the second phases become homogeneously dispersed, the particles can hinder/block further grain rotation, being easier when the volume fraction is very high, i.e. in CP3-MgNi alloy.

In the case of PM alloys the mechanism is simpler. As expected from the homogeneity of the fine dispersion of second phases in a fine-grained magnesium matrix (around 0.5 μm) and the absence of coarse non-recrystallized regions, the conditions for superplasticity should coincide with the last stage of deformation in CP alloys. Since the grain size of the alloy is smaller, an improvement in the superplastic behaviour could be expected for PM alloys compared to CP alloys. In fact, the elongation to failure of both PM alloys at 250°C is already twice that of CP alloys. Even, at 300°C, the maximum elongation of all materials corresponds to the PM-MgNi2 alloy (around 400%). At higher temperatures, however, the elongation tends to decrease gradually in such a way that both PM alloys do not behave superplastically. The microstructure after deformation is identical to that of non-deformed samples, and just some grain coarsening up to sizes about 1-1.5 μm is observed at 350 and 400°C. According to $\dot{\varepsilon}$-σ plots the superplastic range, with $n\approx2$, is shifted to higher strain rates that in the case of CP alloys. At 300°C, the interval in which $n\approx2.5$ is comprised between 10^{-4} to 10^{-2} s^{-1}, and at 350°C and 400°C $n\approx2$ at strain rates higher than 10^{-3} and 10^{-2} s^{-1}, respectively. Consequently, both PM alloys are outside the superplastic regime when tested at 10^{-4} s^{-1} at 350 and 400°C. An increase in the strain rate should be enough to attain superplastic conditions. The tendency of PM alloys to develop cavities is an indication of inadequate accommodation of the dislocations in regions where grain rotation is occurring. It is interesting to note that the number of cavities as well as their size are higher for the PM-MgNi2 alloy than for PM-MgNi3 alloy. This suggests that deformation in the PM-MgNi2 alloy can proceed with a higher volume fraction of cavities while a lower volume fraction of them leads to premature failure in the PM-MgNi3 alloy. This implies that elongation depends on the volume fraction of second phases. An optimal volume fraction of second phases is required to accommodate rotation of grains as well to restrict grain coarsening in the course of superplastic flow. A higher volume fraction could make harder grain rotation, preventing further rotation or even favouring cavity formation.

5. Conclusions

From this research, the following conclusions can be drawn:

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1. Below 200°C, the highest yield stress values correspond to the alloys with higher nickel contents. This is indicative that the yield stress depends more on the composition of the alloy than in the processing route, although for a given composition the maximum value is achieved in PM alloys.

2. Mechanical strength of CP alloys is due to the combined effect of fine grain size and significant load transfer from the magnesium grains towards the hard second phase particles. Inferior grade of aggregation in second-phase arrangements of the MgNi2 alloy compared to the MgNi3 alloy favours a more effective load transfer.

3. The mechanical strength of PM alloys is due to their fine grain size, smaller than 1 μm, and the hardening effect of very fine second phase particles.

4. Independently of the composition and/or the processing route followed to prepare the alloys, the strength of the alloys above 200°C drops to very low values although they exhibit superplastic behaviour.

5. Superplasticity of CP alloys proceeds through a complex mechanism involving grain boundary/interface sliding from the initial stages of plastic deformation, recrystalllization of coarse–grained areas, multiple cracking of coarse second phases and their subsequent redistribution in the magnesium matrix. The deformation mechanism of PM alloys is simpler and it coincides with the last stage of deformation in CP alloys.

Acknowledgements

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Table Captions

Table 1. Values of *n* for all MgNi2 and MgNi3 alloys depending on the temperature and strain rate.

Figure Captions

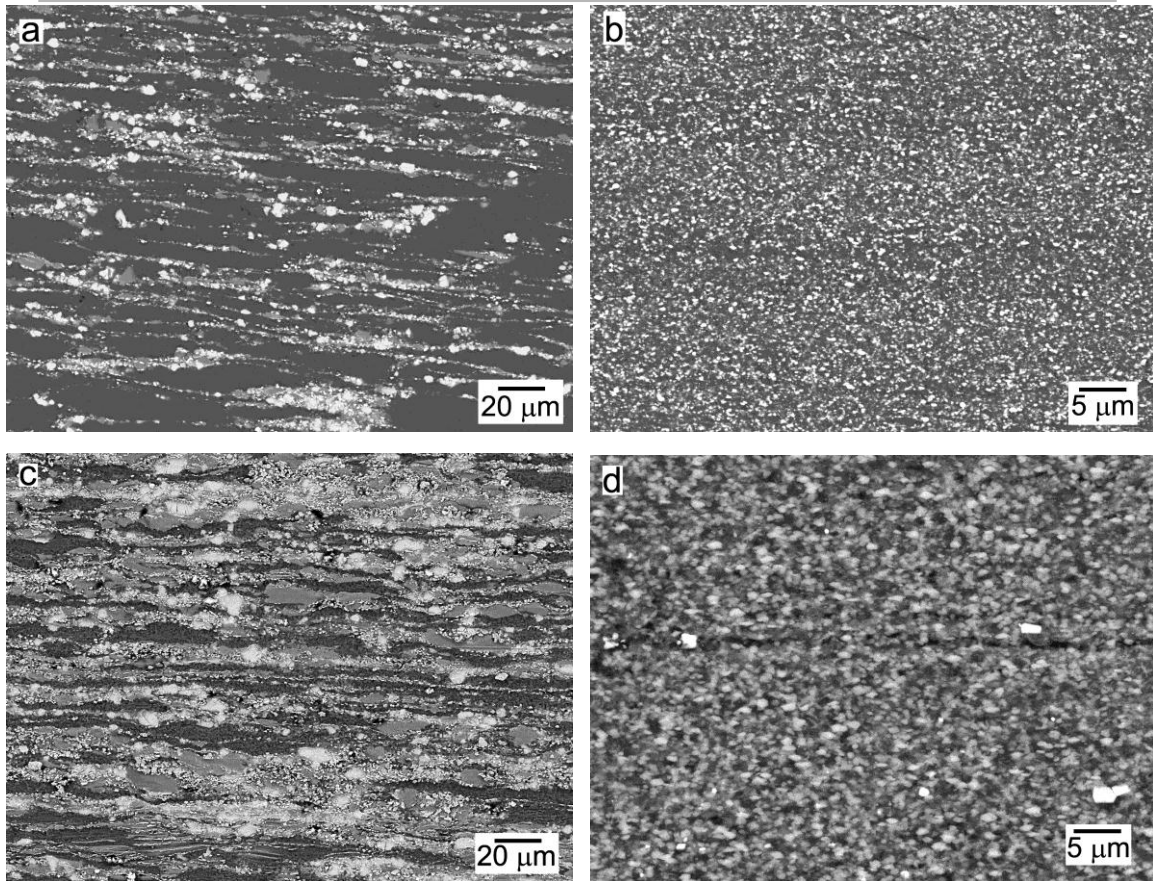Fig. 1. Backscattered SEM images of the microstructures along the extrusion direction of the alloys extruded at 400°C. (a) CP-MgNi2, (b) PM-MgNi2, (c) CP-MgNi3, (d) PM-MgNi3.
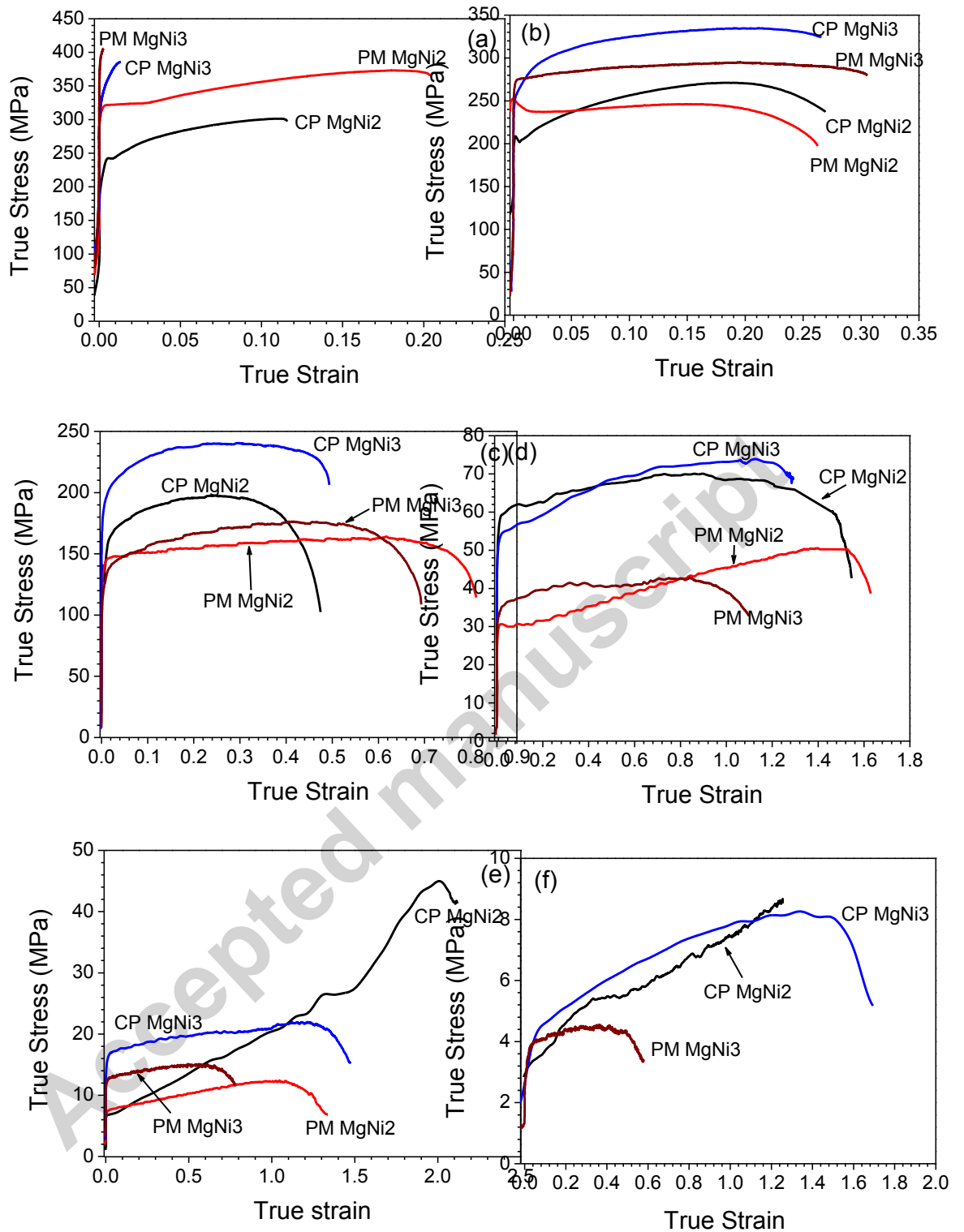
Fig. 2. True stress - true strain curves for all materials at different temperatures. (a) 100°C, (b) 200°C, (c) 250°C, (d) 300°C, (e) 350°C, (f) 400°C.
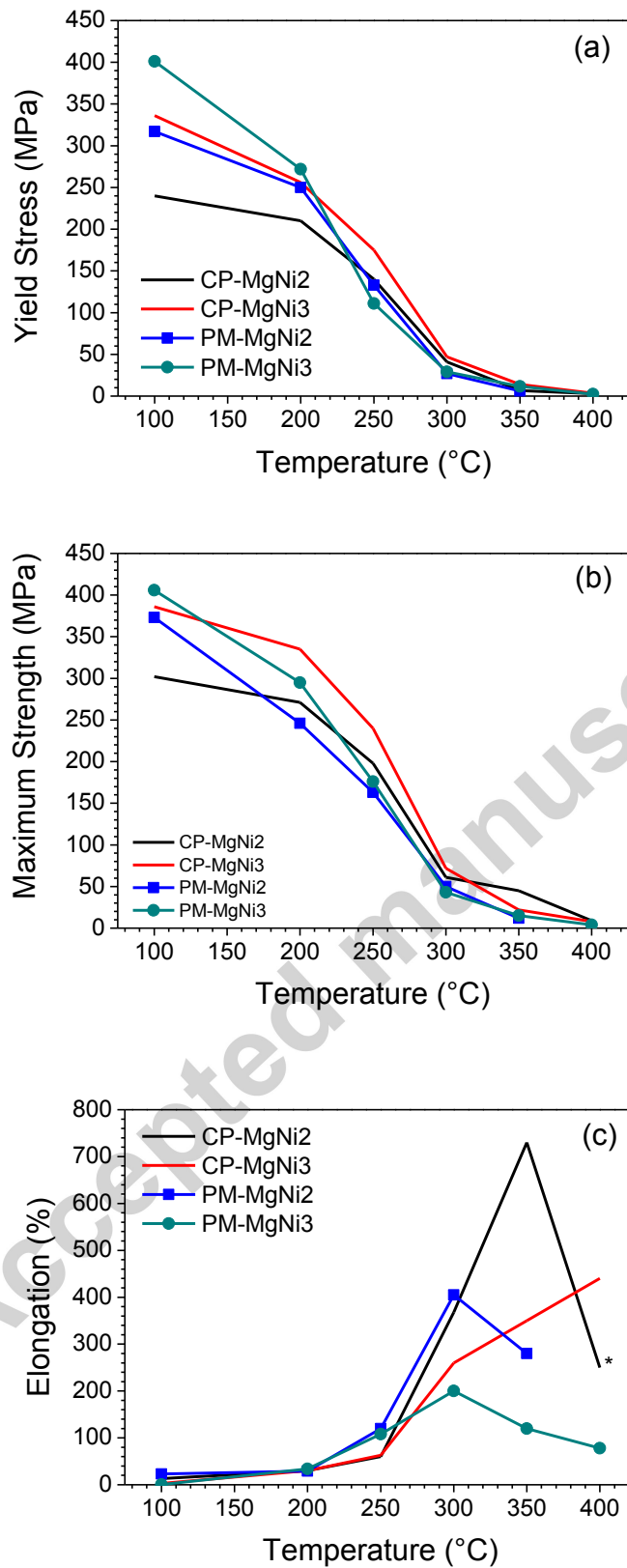
Fig. 3. Evolution of the mechanical properties for all alloys in the 100-400°C temperature range. (a) Yield stress, (b) maximum strength, (c) elongation to failure.

Fig. 4. Strain rate–true stress plot at different temperatures for CP and PM alloys. (a) 100°C, (b) 200°C, (c) 300°C, (d) 350°C, (e) 400°C.

Fig. 5. Microstructure of the longitudinal section for the CP-MgNi2 alloy deformed to failure at 200°C. (a) Outside the necked region, (b) inside the necked region.

Fig. 6. Microstructural features observed in the longitudinal section of the PM-MgNi2 alloy deformed to failure at 200°C: (a) Voids close to the fracture surface, (b) absence of cracking in second phases outside the necked region, (c) numerous cracked second phases near the fracture surface. White arrows mark voids while red arrows show fragmented particles.

Fig. 7. Grain structure along the longitudinal section of the PM-MgNi2 alloy deformed to failure at 200°C: (a) Region close to the fracture surface, (b) within the necked region, (c) outside the necked region, (d) outside the gauge length in the head of the sample.

Fig. 8. Microstructure along the longitudinal section of the samples tested at 250°C. (a) CP-MgNi3 sample deformed 8 %, (b) region close to fracture of CP-MgNi2 sample, (c) within the necked region in CP-MgNi2 sample deformed to failure, (d) outside necked region in CP-MgNi2 sample deformed to failure, (e) head of the CP-MgNi2 sample deformed to failure, (f) within the necked region in CP-MgNi3 sample deformed to failure. Insets show the grain structure of the magnesium matrix revealed by chemical etching.

Table 1. Values of the stress exponent n between 100 and 400°C for all MgNi2 and MgNi3 alloys.

T (°C)	CP-MgNi2		PM-MgNi2		CP-MgNi3		PM-MgNi3	
	n	$\dot{\varepsilon}\ (s^{-1})$	n	$\dot{\varepsilon}\ (s^{-1})$	n	$\dot{\varepsilon}\ (s^{-1})$	n	$\dot{\varepsilon}\ (s^{-1})$
100	22	10^{-4}-10^{-3}	35	10^{-5}-10^{-2}				
200	46	10^{-4}-3×10^{-2}	29	10^{-5}-10^{-2}	18	10^{-5}-10^{-2}	16	10^{-4}-3×10^{-2}
300	7 2	$>3\times10^{-3}$ $<3\times10^{-4}$	>5 2.5 >6	$>3\times10^{-3}$ 3×10^{-4}-3×10^{-3} $<3\times10^{-5}$	>5 2.5	$>10^{-3}$ $<10^{-3}$	>8 3 >5	$>3\times10^{-2}$ 10^{-4}-10^{-2} $<10^{-4}$
350	2	3×10^{-5}-3×10^{-3}	2 8	$>3\times10^{-3}$ $<10^{-4}$	2.5	10^{-5}-10^{-2}	2 4	$>3\times10^{-2}$ $<3\times10^{-3}$
400	2	10^{-4}-3×10^{-2}			2 4.5	3×10^{-3}-3×10^{-2} 3×10^{-5}-3×10^{-4}	2 7	$>3\times10^{-2}$ $<10^{-3}$

Fig. 1. Backscattered SEM images of the microstructures along the extrusion direction of the alloys extruded at 400°C. (a) CP-MgNi2, (b) PM-MgNi2, (c) CP-MgNi3, (d) PM-MgNi3.

Fig. 2. True stress - true strain curves for all materials at different temperatures. (a) 100°C, (b) 200°C, (c) 250°C, (d) 300°C, (e) 350°C, (f) 400°C.

Fig. 3. Evolution of the mechanical properties for all alloys in the 100-400°C temperature range. (a) Yield stress, (b) maximum strength, (c) elongation to failure.
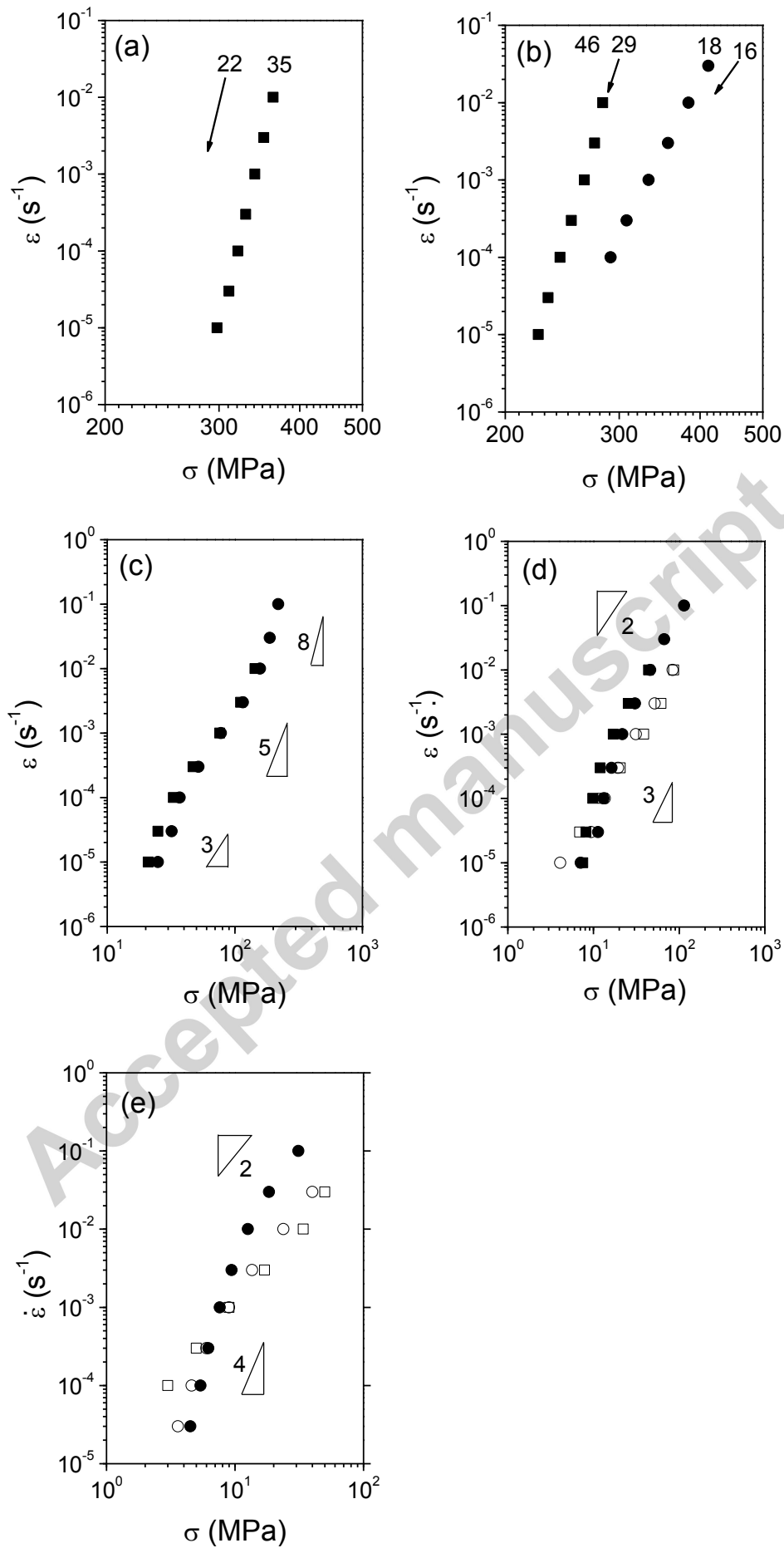
Fig. 4. Strain rate–true stress plot at different temperatures for CP and PM alloys. (a) 100°C, (b) 200°C, (c) 300°C, (d) 350°C, (e) 400°C. (□ CP-MgNi2, ■ PM-MgNi2, ○ CP-MgNi3, ● PM-MgNi3)

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Fig. 5. Microstructure of the longitudinal section for the CP-MgNi2 alloy deformed to failure at 200°C. (a) Outside the necked region, (b) inside the necked region.

Fig. 6. Microstructural features observed in the longitudinal section of the PM-MgNi2 alloy deformed to failure at 200°C: (a) Voids close to the fracture surface, (b) absence of cracking in second phases outside the necked region, (c) numerous cracked second phases near the fracture surface. White arrows mark voids while red arrows show fragmented particles.

Fig. 7. Grain structure along the longitudinal section of the PM-MgNi2 alloy deformed to failure at 200°C: (a) Region close to the fracture surface, (b) within the necked region, (c) outside the necked region, (d) outside the gauge length in the head of the sample.

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Fig. 8.   Microstructure along the longitudinal section of the samples tested at 250°C. (a) CP-MgNi3 sample deformed 8 %, (b) region close to fracture of CP-MgNi2 sample, (c) within the necked region in CP-MgNi2 sample deformed to failure, (d) outside necked region in CP-MgNi2 sample deformed to failure, (e) head of the CP-MgNi2 sample deformed to failure, (f) within the necked region in CP-MgNi3 sample deformed to failure. Insets show the grain structure of the magnesium matrix revealed by chemical etching.

Fig. 9. Regions close to the fracture surface in the samples tested at 250°C. (a) PM-MgNi2, (b) PM-MgNi3 alloy. Red arrows illustrate some examples of cavities.
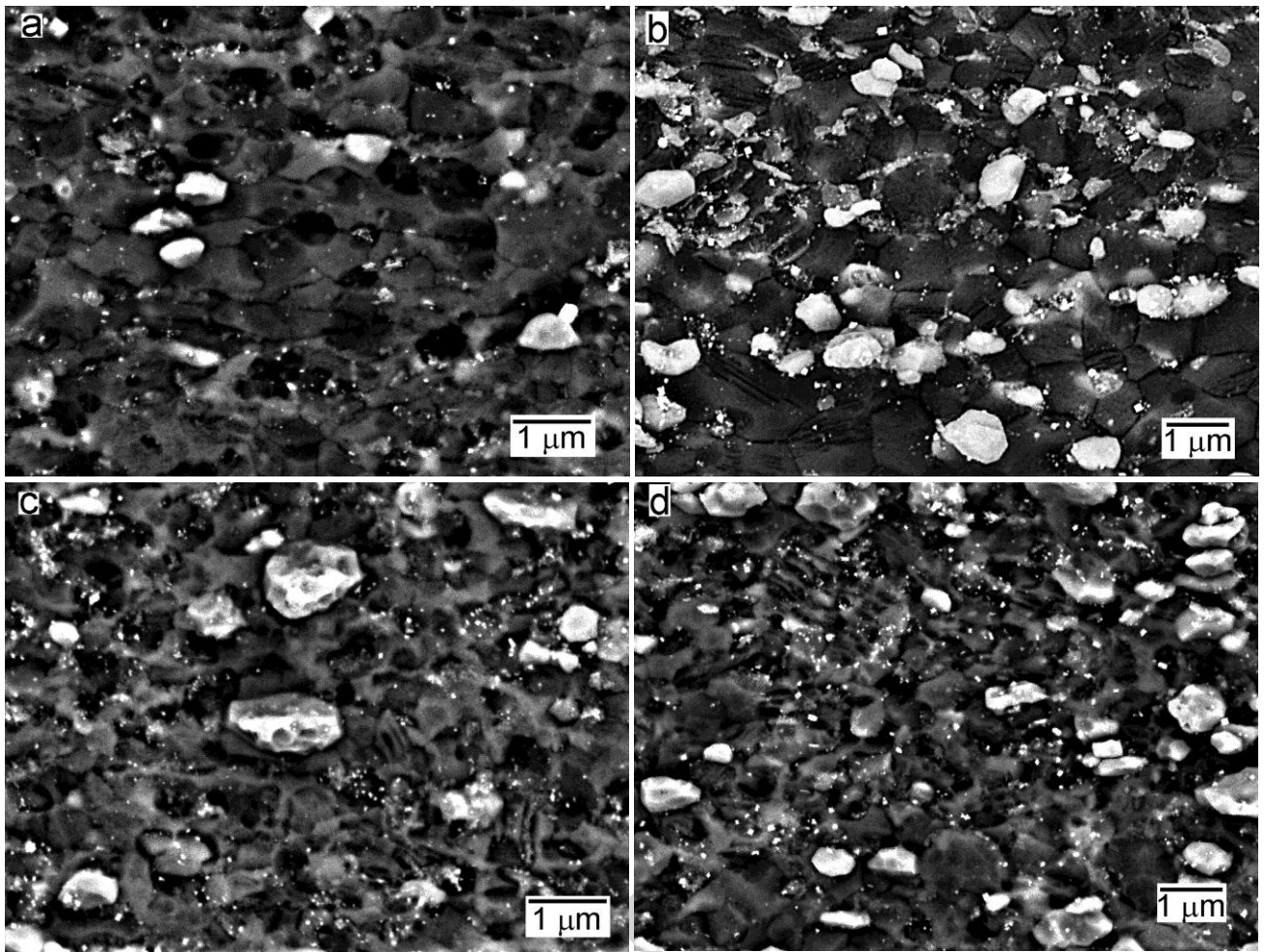
Fig. 10. Microstructure along the longitudinal section of the samples deformed to failure at 250°C. (a) PM-MgNi2 (gauge length), (b) PM-MgNi2 (head), (c) PM-MgNi3 (gauge length), (d) PM-MgNi3 (head).
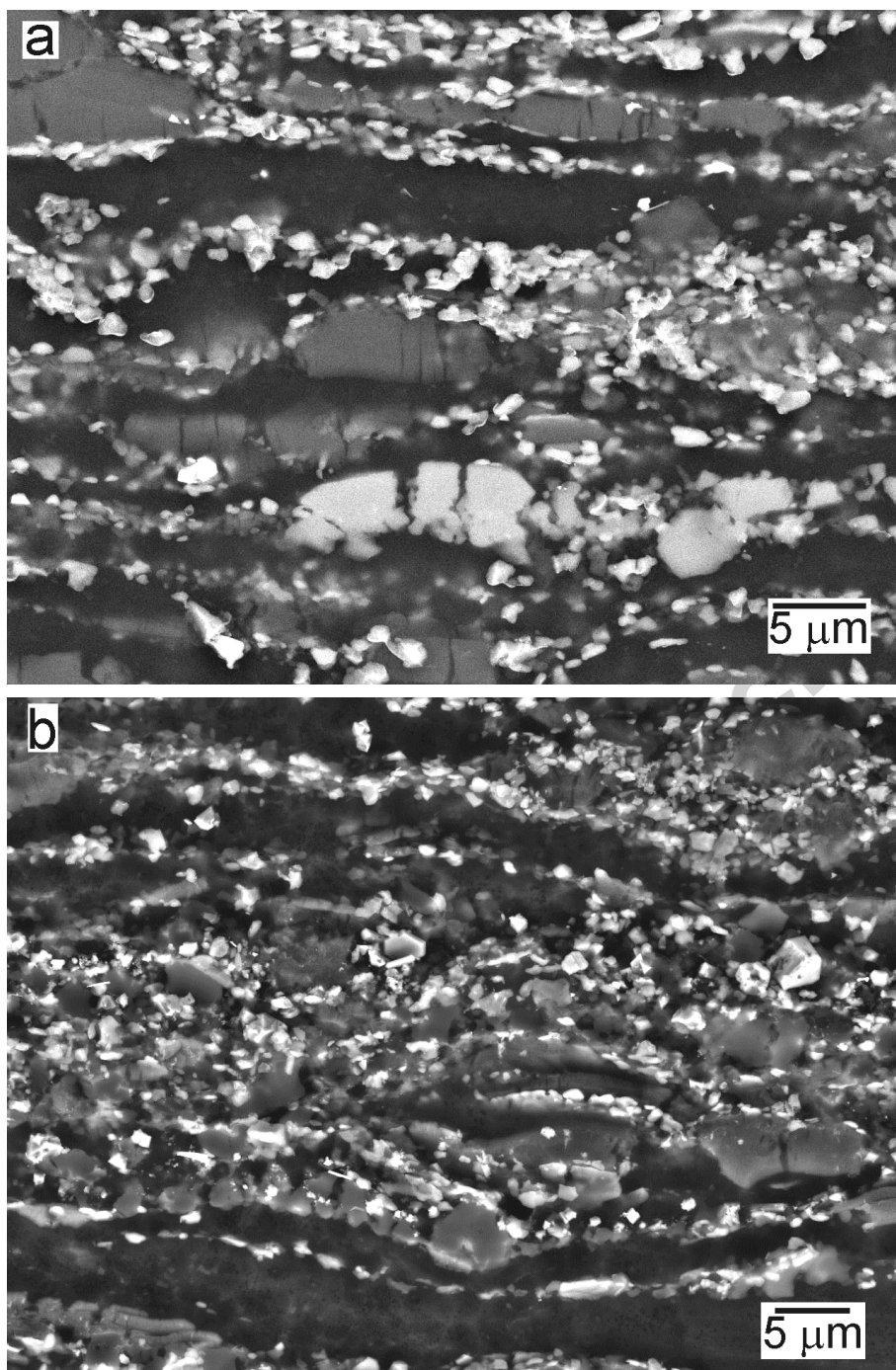
Fig. 11. Fracture of second phases during plastic deformation of CP-MgNi3 alloy.
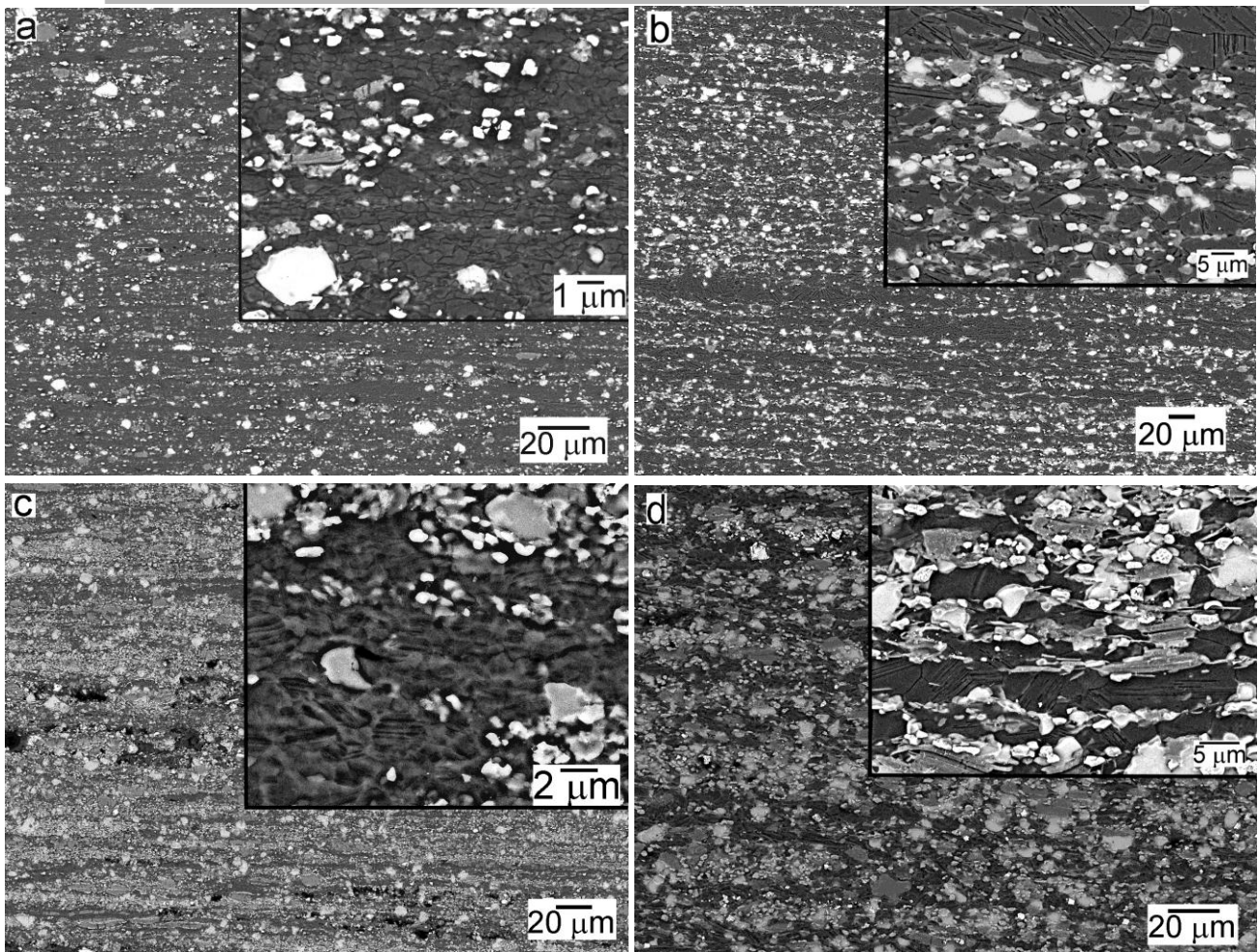(a) Sample deformed 8 % at 300°C, (b) sample deformed 40 % at 350°C.

Fig. 12. Longitudinal sections showing the redistribution of second phases during superplastic deformation: (a) CP-MgNi2 alloy deformed to failure at 300°C (b) CP-MgNi2 alloy deformed 250 % at 400°C, (c) CP-MgNi3 alloy deformed to failure at 300°C, (d) CP-MgNi3 alloy deformed to failure at 400°C. The insets reveal the grain structure in the deformed regions.
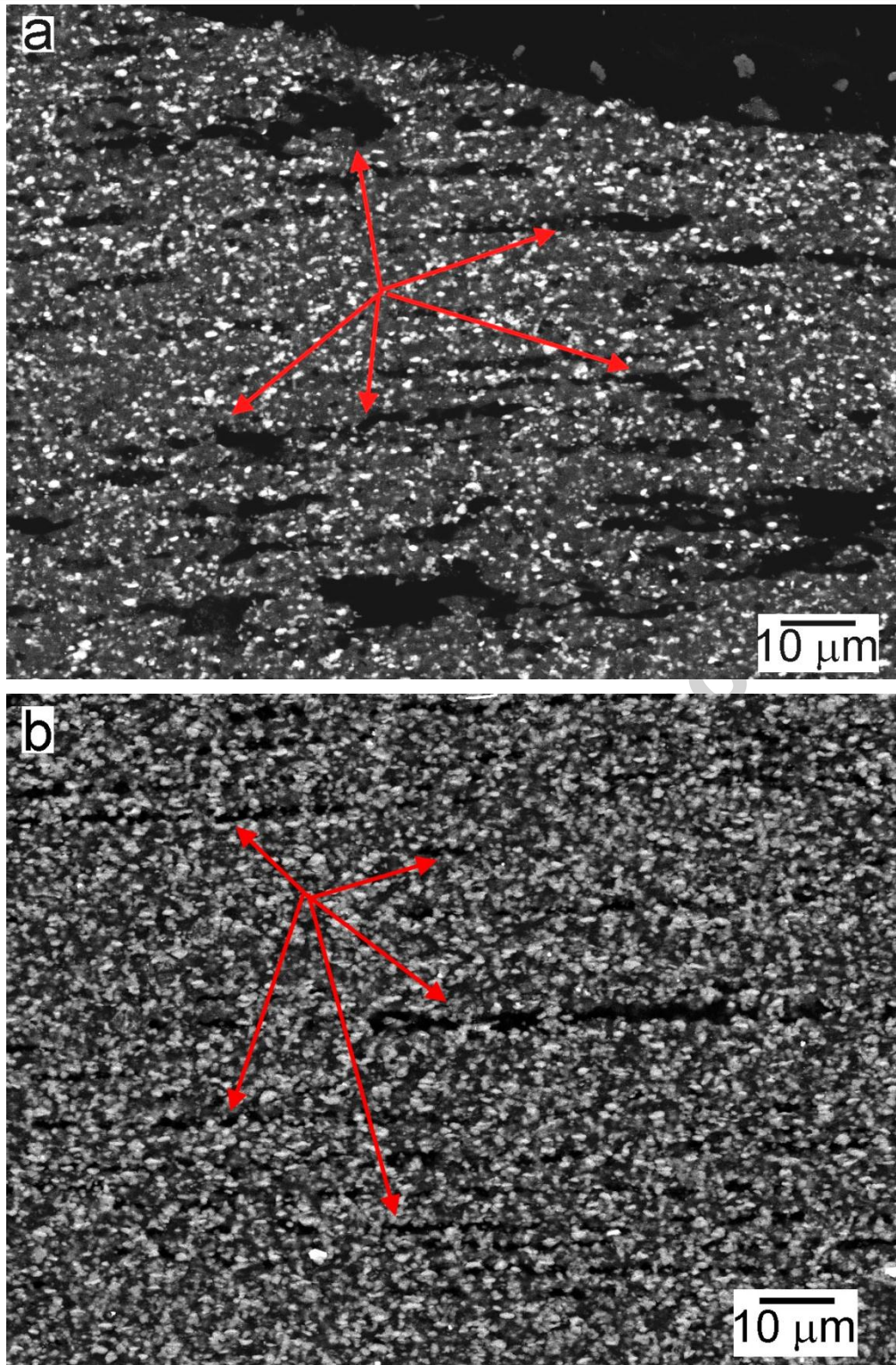
Fig. 13. Cavities developed in regions near the fracture surface in PM alloys deformed at 350°C. (a) PM-MgNi2 alloy, (b) PM-MgNi3 alloy.

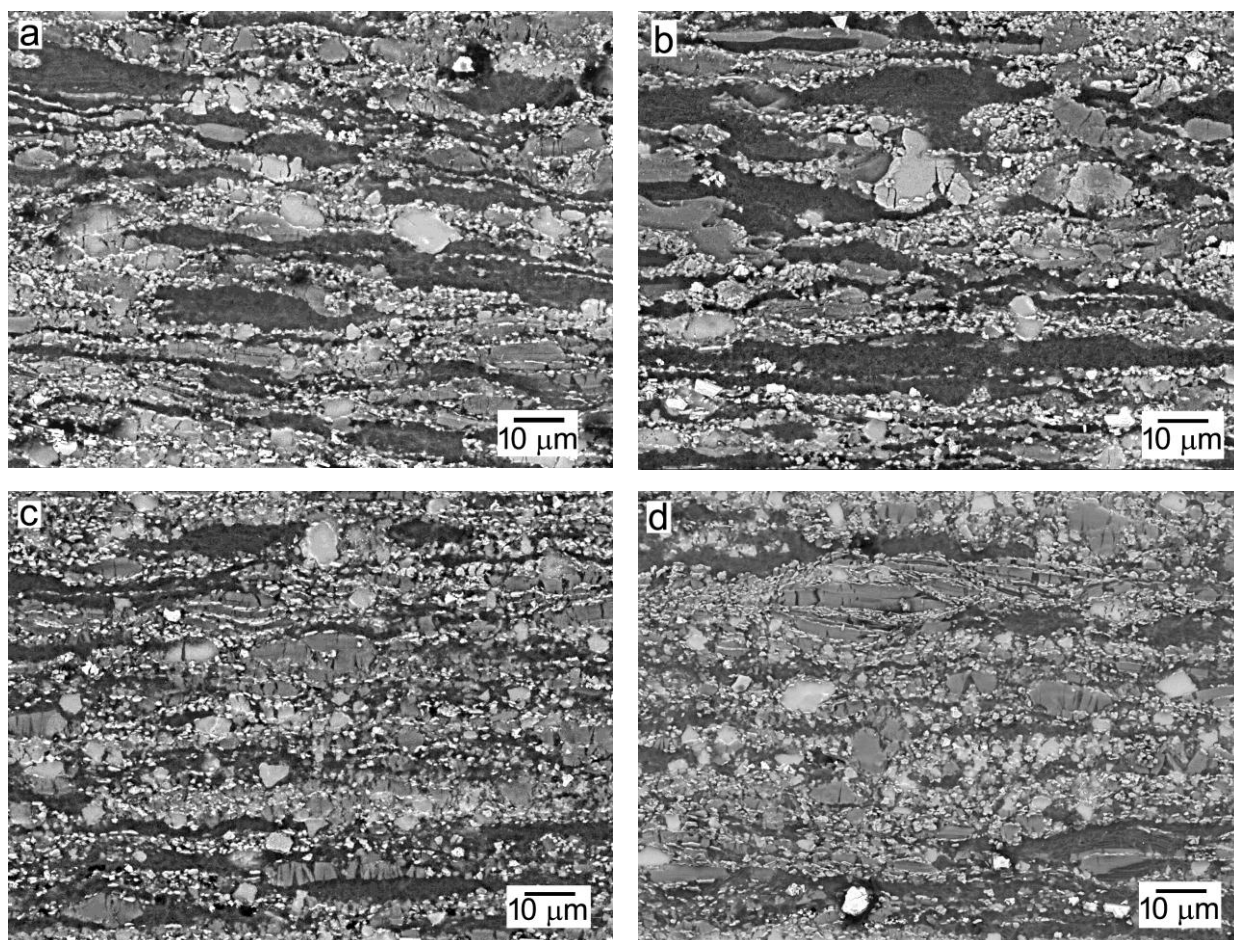Fig. 14. Microstructure of MgNi3 samples tensile tested at 300°C. (a) 30 % of elongation, (b) 90 % of elongation, (c) 150 % of elongation, (d) up to failure.