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Twinning Assisted Crack Propagation of Magnesium-Rare Earth Casting and Wrought Alloys under Bending

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Abstract. Due to their high specific strength, good corrosion resistance and high temperature strength Magnesium alloys containing Rare Earth additions are promising candidates for structural and engine applications in the transportation industry. Also medical applications, like bone screws and nails, benefit from their moderate corrosion rate and biocompatibility. All applications need materials which show a high strength, ductility and fracture toughness in case a crack has formed during service to keep safety against rupture. In this study four extruded Mg10Gd based alloys modified with Nd and La have been 3-point-bend tested at low a deformation speed to evaluate the influence of the microstructure on crack growth. A comparison to the cast material (subjected to T4 to increase ductility and to reduce the dendritic microstructure) shows an increase in strength and ductility due to the fine grained microstructure as a result of recrystallization during extrusion. The maximum bending strength and outer strain to crack initiation is also strongly influenced by the alloying system itself. The influence of Nd and La to the binary alloy Mg10Gd is discussed in using tensile, compression and bending tests. The increase in strength results in reduced elongation to fracture in tension loading as well as the outer strain for the crack initiation during bending tests. Tensile tests are often discussed to be not a reliable method for determining the Young's modulus of Magnesium. Therefore resonance frequency damping analysis has been applied to determine the dynamic modulus of elasticity, which is compared with the flexural (bending) modulus. Crack growth is discussed using light microscopy and correlated with bending stress-strain curves. The crack growth rate of the extruded, fine grained material is many times higher than of the cast, coarse grained material. Crack propagation is mostly transgranular and assisted by twinning.

Introduction

Rare Earth (RE) elements are useful in Mg alloys when creep resistance and moderate corrosion rate is required [1]. The best corrosion resistance and strength combination among binary Mg-Gd alloys has been found in Mg10Gd [2]. To increase the creep, fatigue and corrosion properties even more other elements, like Y, Zr or Nd are added to the binary Mg10Gd system. A review by Mirza et al. [3] of RE containing Mg alloys on the basis of Mg with higher Gd content for fatigue studies presented promising results, like effectively weakened crystallographic textures, suppressed twinning and enhanced fatigue strength. During hot extrusion most of the Gd-containing intermetallics break and then disperse along the direction of hot extrusion. Without further heat treatment the particles have a rather large particle size. The influence of these particles as well as the suppression of twinning due to the presence of RE-rich precipitates and finer grain size compared with cast condition on the crack propagation is of interest in this study. The role of modifying the alloys with addition of Nd and/or La on the crack propagation in Mg10Gd is also investigated.

Experimental procedure

Processing. Cast Mg10GdxNdxLa alloys investigated were prepared at the HZG using permanent mold direct chill casting. The technique itself and its processing parameters can be found in previous work [8,9]. Gd, Nd and La were added in terms of pure elements to the Mg melt before casting. The cast Mg10GdxNdxLa alloys were annealed and then extruded at the Extrusion Research and Development Center, TU Berlin. The materials (bars) were extruded at an overall temperature of 420 °C. A product speed of 2.2 m/min and an extrusion ratio of 37 were used.

Testing. The overall chemical composition was analyzed with inductively coupled plasma–optical emission spectroscopy (ICPOES). The cast material was solution heat treated (T4) at 525 ^{o}C for 24 h and quenched into water at room temperature to increase ductility. The mechanical properties were measured at room temperature with an initial strain rate of $1x10^{-3}$ mm s^{-1}. 5 to 10 samples were tested for each alloy. Since tensile testing is not a reliable method for determining the Young's modulus of Mg alloys, resonance frequency damping analysis was used to determine the dynamic modulus of elasticity (DME). Samples were cut with a hack sawing machine and the surface were then milled and ground to reduce residual stresses for Vickers hardness testing. The three-point bending tests were conducted using a span of 90 mm (cast material with geometry of 110x20x 4/6 mm^3) and 40 mm (extruded material 70x10x8 mm^3). The diameter of the support brackets and plunger were 10 mm and a deformation speed of 1 mm/min was used during the tests. The plates were tested in the upright position to guarantee longer crack propagation. The deflection during bending was monitored and converted into outer strain (strain at the outer surface of the sample) in dependence of bending stress. Metallographic investigations were conducted according to Kree [4].

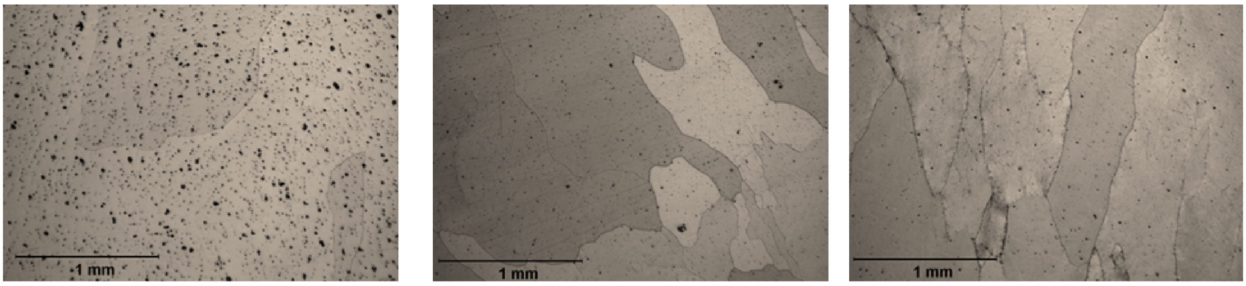
Results and Discussion

Materials and Microstructure. The chemical compositions of all 7 alloys are shown in Table 1. The actual Gd, Nd and La content is less than the nominal 10 and 1 wt.% and differs slightly. Fig. 1 shows that cast material is very coarse grained and with the addition of Nd the grain size decreases. A rather small grain size develops by dynamic recrystallization during extrusion (see microstructure of the extruded alloys in Fig. 2). With addition of Nd and/or La the average grain size of the extruded alloys decreased and the volume fraction of the second phase increased. These phases act as nuclei during the recrystallization process or restrict grain growth, where particles at the grain boundaries hinder the boundary mobility (growth restriction mechanism). This explains the inhomogeneous microstructure; see grain size distribution given with grain size in table 2. Grains near intermetallics show smaller grain sizes (less than 10 μm) compared to regions with lower amount of intermetallics. The intermetallics align along the extrusion direction.

Table 1. Chemical composition in wt. %, grain sizes in μm and second phases.

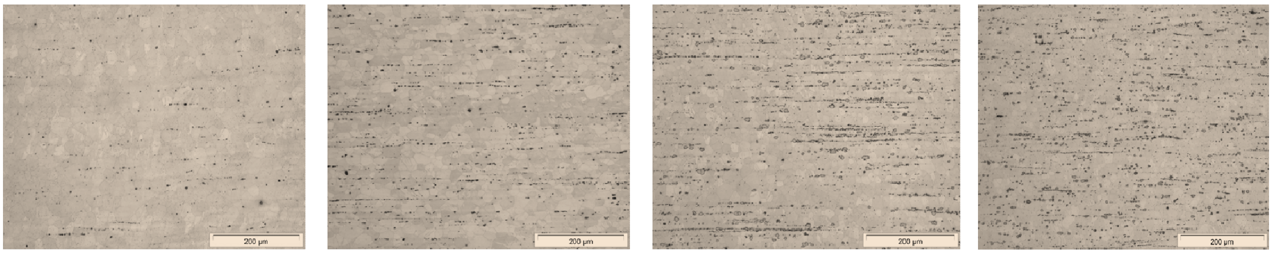
		Gd	Nd	La	grain size	second phases
Mg10Gd	cast-T4	9.20	-	-	1200 ± 715	Mg_5Gd
Mg10Gd1Nd		8.13	0.74	-	925 ± 695	$Mg_5(Gd,Nd)$
Mg10Gd2Nd		9.17	1.75	-	775 ± 520	$Mg_5(Gd,Nd)$
Mg10Gd	extruded	9.70	-	-	24 ± 11.8	Mg_5Gd
Mg10Gd1Nd		9.80	0.88	-	17 ± 8.3	$Mg_5(Gd,Nd)$
Mg10Gd1La		9.70	-	0.83	15 ± 6.5	$Mg_5(Gd,La)$
Mg10Gd1Nd1La		9.50	0.88	0.89	13 ± 5.6	$Mg_5(Gd,Nd,La)$

Mg-alloys containing more than 5 wt.% Gd, Mg_5Gd intermetallics can be observed mostly on grain boundaries [2], as shown in table 1. The main intermetallic phase in all alloying systems is Mg_5Gd. In alloys containing Nd and/or La, Gd is substituted, but the crystal structure persists. Nd and La seem to accelerate the growth of intermetallic phases. With additions of Nd and/or La the particle size as well as the volume fraction of particles increased significantly (see micrographs in figure 2). The largest intermetallic particles can be found at the grain boundary triple points. At these points the distance between Mg base alpha phase grains is large, allowing the growth of the particle.



a) b) c)

Fig. 1. Microstructure of cast Mg10GdxNd in T4 condition: a) Mg10Gd, b) Mg10Gd1Nd and c) Mg10Gd2Nd.



a) b) c) d)

Fig. 2. Microstructure of extruded a) Mg10Gd, b) Mg10Gd1Nd, c) Mg10Gd1La and d) Mg10Gd1Nd1La.

Mechanical Properties. The mechanical properties are given in table 2. The cast-T4 material shows a lower strength and ductility compared with the extruded material. The addition of Nd and La generally improves the strength compared with the binary Mg-Gd alloy. The increase in the alloying elements in the extruded material results in decrease of ductility in tension and compression. In contrast, the cast-T4 Mg10Gd1Nd alloy shows the highest elongation in tension. Recent publication [5] also reported the highest elongation for as-cast and T6 heat treated Mg10Gd1Nd. However, the compression strain increased for cast-T4 material with increased amount of alloying elements. Microhardness, being more dependent on the area of indentation, shows increased hardness with increase in the alloying elements. The binary Mg10Gd shows lower hardness in the cast-T4 condition in comparison with the extruded condition. The Mg10Gd1Nd shows no difference in hardness. The dynamic modulus of elasticity (DME) also increases with increased alloying addition.

Table 2. Mechanical Properties

		TYS in MPa	UTS in MPa	El. in %	CYS in MPa	UCS in MPa	CS in %	DME in GPa	HV1
Mg10Gd	cast-T4	68.7 ± 2.4	111.7 ± 9.3	3.2 ± 0.5	81.0 ± 3.5	222.8 ± 10.5	17.4 ± 2.1	43.71 ± 0.38	59.0 ± 2.4
Mg10Gd1Nd		108.3 ± 3.4	174.4 ± 0.8	7.0 ± 1.2	59.8 ± 8.6	278.2 ± 7.4	18.2 ± 1.3	44.22 ± 0.15	70.2 ± 4.7
Mg10Gd2Nd		106.4 ± 2.4	162.1 ± 5.8	2.9 ± 0.4	68.1 ± 16.3	276.1 ± 11.1	22.3 ± 3.8	44.45 ± 0.15	72.8 ± 5.1
Mg10Gd	extru.	131.4 ± 2.2	248.9 ± 2.7	22.9 ± 2.8	133.7 ± 0.4	399.2 ± 10.2	26.1 ± 1.3	44.18 ± 0.09	66.8 ± 3.5
Mg10Gd1Nd		138.2 ± 3.3	256.0 ± 4.1	17.3 ± 1.2	148.9 ± 5.7	397.5 ± 14.2	16.8 ± 1.2	44.95 ± 0.05	70.6 ± 6.3
Mg10Gd1La		149.2 ± 8.4	248.0 ± 3.9	17.0 ± 3.7	149.4 ± 1.1	395.8 ± 12.5	18.4 ± 0.5	44.57 ± 0.04	75.0 ± 4.6
Mg10Gd1Nd1La		165.4 ± 0.9	253.4 ± 2.2	12.2 ± 1.1	161.2 ± 1.3	393.4 ± 23.3	13.0 ± 2.1	45.01 ± 0.04	77.4 ± 4.9

Crack propagation under 3-point-bending of cast and extruded Mg10GdxNd. Firstly the influence of extrusion on the 3-point-bending properties and crack propagation in comparison with the cast-T4 material is of interest. Bending tests of Mg10GdxNd were conducted until the stress sustained was 10% of the maximum stress. Fig. 3 shows a significant increase in maximum stress reached under bending for the extruded, fine grained material. Crack initiation (followed by crack growth) took place after bending stress is beyond its maximum. Generally, the binary alloy Mg10Gd shows the highest elongation at fracture for a given processing condition. Even though the strength and strain at fracture can be improved by extrusion, there are very high stress drops after crack initiation indicating increased crack growth rates. The coarse grained cast-T4 material twinned heavily during crack propagation (strong acoustic emissions was audible by ear). There was no audible noise during 3-point-bend testing of the fine grained extruded material (increased amount of particles suppress twinning). Stress reduction to 10% of the maximum stress was reached within 10 seconds. In contrast, in the cast-T4 material crack propagation lasted up to 200 seconds. If however, the area underneath the bending stress - outer strain curve was considered to be the toughness, extruded material shows much higher toughness than the cast-T4 condition, which was expected by the higher tensile and compressive strength and ductility.

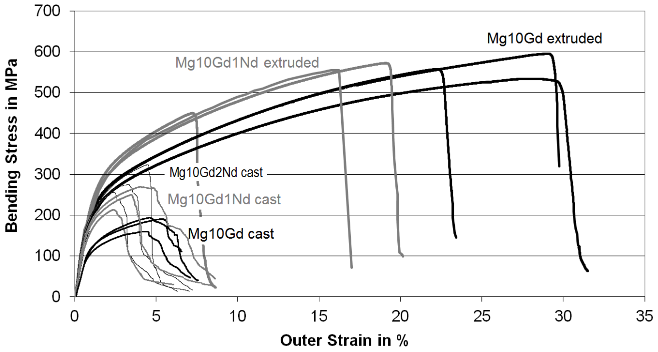


Fig. 3. Bending Stress - Outer Strain curves of cast and extruded Mg10Gd and Mg10Gd1Nd and cast Mg10Gd2Nd

Fig. 4 underlines the different behavior in crack growth within the coarse grained microstructure of the cast and solution heat treated material in comparison with the extruded material (much smaller grain sizes). The micrograph in Fig. 4a shows a strong crack reorientation (zigzag) in the cast-T4 Mg10Gd1Nd. In contrast the extruded Mg10Gd1Nd shows a crack that propagates more along a straight line (Fig. 4b). Higher magnification images of the crack in the cast-T4 material reveals the interaction of the crack with twin boundaries (Fig. 4c), which seem to "carry" the crack over to grain boundaries. Detailed information on this behavior is discussed in detail in [6]. The interaction between crack and twins, grain boundaries and intermetallic particles in the fine grained microstructure is more complex (Fig. 4d): The crack propagation is primary influenced by the twin boundaries (compare crack direction and twin boundary orientation within the same or other grains). Additionally, micro-cracks are initiated by twin boundaries itself as seen the small cracks of 10 to 20 µm away from main crack in parallel to the twin boundaries nearby on Fig. 4d. Grain boundaries act primary as a barrier to further crack growth. The crack propagation is mainly trans-granularly and influenced by the stress fields of intermetallic particles. Hence, the weak interfaces between matrix-particle, cracked brittle second phases or twin boundaries all initiate micro-cracks and contribute to propagation of the main crack. This gives the crack its direction.

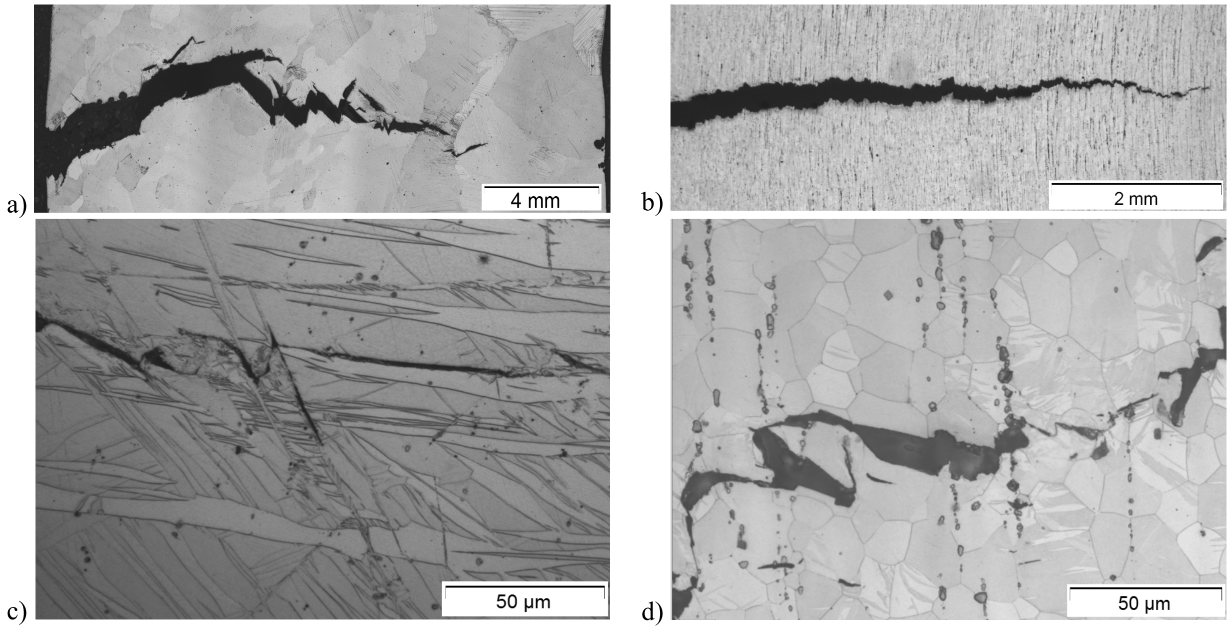


Fig. 4. Micrograph of crack propagation under bending stress in Mg10Gd1Nd: a and c: cast-T4 Mg10Gd1Nd, revealing strong crack redirection, b and d: extruded Mg10Gd1Nd, showing rather straight line in crack growth (overview in a/b and detailed interaction with grain boundaries, twins and second phases in c/d).

Crack propagation under 3-point-bending of extruded Mg10GdxNdXLa. Fig. 5 shows bending stress - outer strain curves of all investigated extruded alloys. The bending yield strength increases in ternary and quaternary alloys. However, the strengthening caused by intermetallic phases causes the lower ductility of these alloys. This is due to the large and brittle second phases which increase in the ternary and quaternary alloys. Table 2 shows that the DME increased with increased alloying additions. Looking at the elastic parts of the curves in Fig. 5, the bending modulus follows the same pattern as seen with DME values and Fig. 5 suggests, that the addition of Nd and La has a pronounced effect on the increased elastic moduli within this group of alloys.

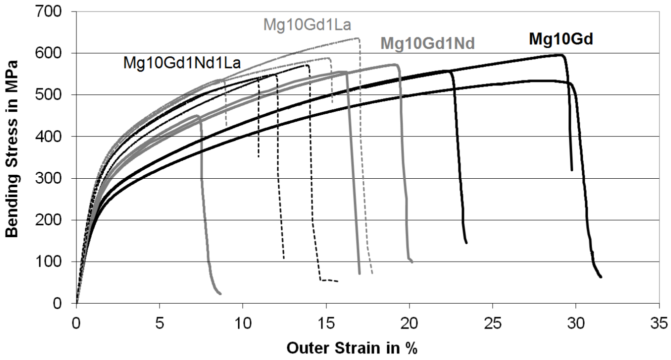


Fig. 5. Bending Stress - Outer Strain curves of extruded Mg10GdxNdxLa.

To evaluate the influence of La additions to the binary Mg10Gd and ternary Mg10Gd1Nd alloy on crack propagation, micrographs of higher magnification especially at the crack tip are used. Fig. 6a and 6c presents the crack paths of the binary Mg10Gd alloy, with the lowest volume fraction of particles, where the crack path is mostly influenced by the twin boundaries (see twin initiated micro-cracks nearby). As already mentioned increased alloying addition increased the volume fraction and the size of second phases, as illustrated in Fig. 6b and 6d for Mg10Gd1La and Fig. 4d for Mg10Gd1Nd and Fig. 7a for Mg10Gd1Nd1La. In the micrographs, Fig. 6b, 6d and 7a, cracks form within the brittle second phases and along the interfaces between Mg base alpha phase grains and intermetallic phases, but less at twin boundaries. It seems the presence of higher volume fraction of intermetallics suppress twinning. Unfortunately the intermetallic phases are larger with the addition Nd and/or La, resulting in brittleness.

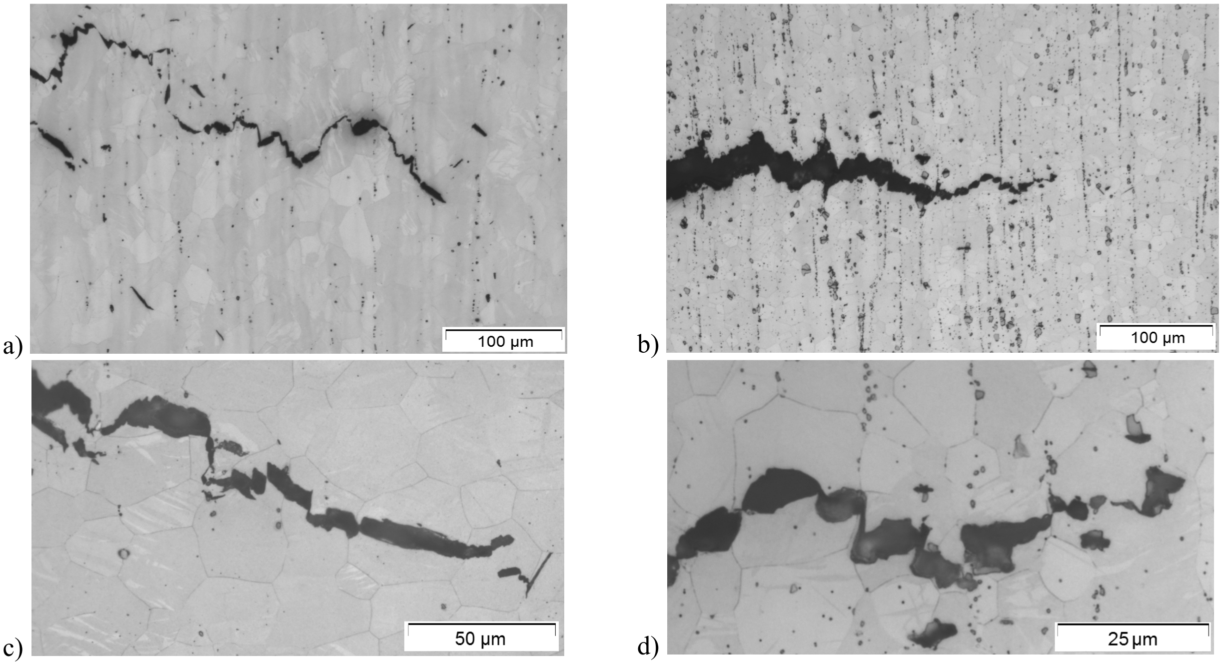


Fig. 6. Micrograph of crack propagation under bending in extruded Mg10Gd and Mg10Gd1La: a and c: extruded Mg10Gd, revealing twin boundaries as crack initiator, also interacting with second phases of low volume fraction, b and d: extruded Mg10Gd1La, showing cracked brittle phases of larger size and higher amount as crack initiation.

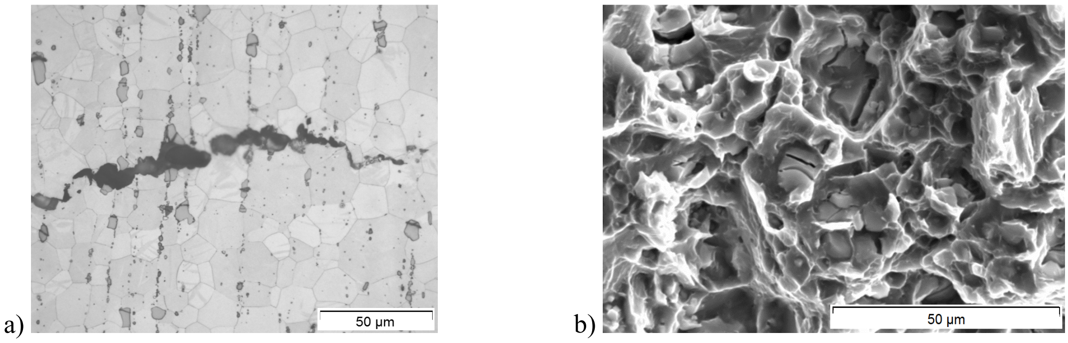


Fig. 7. Micrograph of crack propagation under bending (a) and SEM fracture surface (b) of extruded Mg10Gd1Nd1La, revealing trans-granular ductile fracture with large, brittle cracked second phases nearby and at fractured surface.

The micrograph, Fig. 7a (Mg10Gd1Nd1La: highest volume fraction and size of intermetallics) shows ones more that the large brittle intermetallics initiate cracks (perpendicular to tension stress) and accommodate the crack propagation. Cracked intermetallics are found at the tension side only. Larger intermetallic particles crack first. Research on the critical particle size and stress-strain load limit to crack the intermetallic particles is under progress. The surrounding Mg based matrix material shows ductile fracture behavior (Fig. 7b).

Summary

On the one hand, Nd and/or La increase tensile and bending yield strength significantly, but hardly the maximum strength. The volume fraction and particle size of intermetallic phases increases with increasing alloying elements. Crack propagation in the binary Mg10Gd alloy is mostly driven by twinning (mainly seen in large grain sizes of cast material). Decreased grain size (wrought material) and increased amount and size of second phases by addition of Nd and/or La seems to suppress twinning, but on the other hand crack initiation and propagation is caused by these brittle and coarse second phases. Looking to the future, the effect of heat treatments of the as extruded material to reduce the brittle second phases (solution heat treatment) and to precipitate much smaller second phases for particle hardening (peak ageing) will be investigated. Furthermore research is currently undertaken to understand how intermetallic phases in relation to the rule of mixture (Gd: high solubility in Mg, Nd: low, La: very low) influence the elastic moduli.

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References

[1] L. L. Rokhlin, Magnesium alloys containing rare earth elements – structure and properties, Taylor & Francis, London, UK (2003).

[2] N. Hort et al., Magnesium alloys as implant materials Principles of property design for Mg-RE alloys, Acta Biomater. 6 (2010) 1714-1725.

[3] F.A. Mirza and D.L. Chen, Fatigue of rare-earth containing magnesium alloys: a review, Fatigue Fract. Engng. Mater. Struct. 37 (2014) 831–853.

[4] V. Kree et al., The Metallographical Examination of Magnesium Alloys, Practical Metallography 41 (2004) 233-2346.

[5] P. Maier et al., Tailoring mechanical and corrosive properties of cast Mg10Gd by alloying Nd and heat treatment, Emerging Materials Research, 2/5 (2013) 249-258.

[6] P. Maier et al., Crack Propagation under Bending in Cast Mg10-GdxNd-T4 Alloys, Magnesium Technology, ISBN: 978-1-118-88816-2 (2014) 77-82.