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Study on Microstructure Evolution of Deformed Mg-Gd-Y-Nd-Zr Heat-Resistant Magnesium Alloys after Solid Solution and Ageing

Yu Jianmin[1*)], Zhang Zhimin[1], Zhang Xing[2], Ren Fengli[2], Wu Yaojin[2]
[1]College of Materials Science and Engineering, North University of China, 030051, Taiyuan, China
[2]Engineering Research Center of Magnesium-base Material Processing Technology, Ministry of Education, 030051, Shanxi, China

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Abstract:
The microstructure evolution of Mg - Gd - Y - Nd - Zr heat-resistant magnesium alloy after deformation and T5 or T6 treatment were studied. In thermoplastic deformation, dynamic recrystallization and dynamic precipitation has been taken place at the same time. The dynamic precipitation reduces the recrystallization nucleation driving force in the grain; it will prevent to occur dynamic recrystallization partially. Solid solution temperature was 530^{o}C and hold 4h. Age hardening treatments were performed at 225^{o}C and hold 16h. The alloy showed the comprehensive properties are obviously improved from T6 to T5 heat treatment. After T5 heat treatment the tensile strength of alloy increased to 359.3 MPa, increased by around 48.5%; Elongation is increasing from 5.17% to 6.5%. After peak ageing treatment, the main precipitation is β' phase, the precipitation phase have obvious pinning effect to grain boundary of the alloy, it will prevent the grain growth ageing for a long- time. At the same time, strengthening role of precipitate phase make its strength increased significantly.

Keywords: *microstructure evolution, Mg-Gd-Y-Nd-Zr, magnesium alloys, solid solution, ageing treatment*

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1. Introduction

In the last several decades the awareness of environmental pollution has increased significantly. Light is one of the effective measures to reduce environmental pollution [1]. Magnesium alloy with high specific strength and specific stiffness, used as lightest metal structural materials, are attracting great attention in lightweight design. However, its poor heat resistance and high temperature mechanical properties severely restrict the wide application of magnesium alloys. The addition of Rare earth elements such as Gd, Y to magnesium alloy can effectively improve the room and elevated temperature mechanical properties. At the same time, it can also improve its high-temperature creep and corrosion resistance. In recent years, it has undertaken many studies on effects of heavy rare earth elements to high temperature properties of magnesium alloys. WE54 and WE43 alloys with high room strength and good thermal stability, are considered to be more successful heat-resistant magnesium alloys now. Currently, WE (Mg-Y-Nd) series alloy are widely used in aerospace, because it

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*) **Corresponding author**: minyu889@163.com

has a good cast, age hardening and high-temperature creep resistance [2-4]. Rokhlin [5] and Kamado [6] found that high temperature strength of Mg-20%Gd (mass fraction) is more than traditional heat-resistant magnesium alloy WE54A. However, the alloys density increased with content of Gd, excessive content of Gd will lead to costs are too high, the density of the alloy is higher and low temperature elongation is lower. Y has a higher solid solubility in Mg alloy (12.5%), compared with Gd, the dense of Y is less, and the price is relatively low. Kamado and Rokhlin and Nikitina and others have developed high mechanical properties magnesium alloys such as Mg–10Gd–5Y–0.5Mn alloy and Mg–10Gd–3Y–0.4Zr alloy. These alloys display higher specific strength at both room and elevated temperature and good creep resistance than conventional Mg alloys and WE54. Precipitation hardening is the most common strengthen mechanism used to improve the mechanical properties of oversaturated solid solution in magnesium alloys [7]. The tensile strength of binary Mg-Gd alloys increase with increasing contents of Gd, solid solution hardening and ageing hardening are more prominent. The improvement of mechanical properties is attributed to the excellent age hardening response of Mg–RE alloys. Currently researchers have focus on the mechanical properties optimization of Mg-Gd-Y rare earth magnesium alloy [8, 9]. This paper study on effects of microstructure and mechanical properties of deformed Mg-Gd-Y-Nd-Zr alloy after T5 and T6 heat treatment.

2. Experimental

A billet about φ140mm×50mm were cut from a cast alloy with the nominal composition reported in Tab. I. The cast ingot was homogenization heat treatment on 550^oC+12h before deformation. The billet was deformed by two stages at 500^oC and 450^oCrespectively, and followed cooled by 90~100^oC hot water. The total deformation degree was ε=1.6. After two deformations, the sheet thickness becomes 14mm. Finally, the sample 10mmx10mmx14mm thick slices were cut from deformed sheet for heat treatments. Heat treatment divide into two groups, one group of the samples were solution treated in an argon atmosphere at $530^oC$for 4h. After solution treatment, the sample was quenched in hot water of about 80^oC and subsequently age treated at $225^oC$for 16h. Another group of the samples were aged immediately (T5 heat treatment.).

Tab. I Chemical composition of experiment sample（wt%）

Elements	Gd	Y	Nd	Zr	Mg
Chemical Composition	8.8	2.5	1.5	0.5	Bal.

The heat treated specimens for microstructure analysis were sectioned parallel to the compression axis. The specimens were polished mechanically and etched in a solution consisting of 4% nitric acid alcohol. Microstructure analysis was carried out by using optical microscope (Axio Imager) and scanning electron microscope (SEM). Hardness of samples was measured on the HR-120 Rockwell hardness tester, with a load of 100Kg and duration of 15S. Each sample was measured 7 times, removing a maximum and a minimum value, and taking the average statistics. The tensile tests were performed, according to the standard of GB6397-86, by WDW-E100D electronic universal testing machine.

3. Results and discussion

3.1 Microstructure before heat treatment

Fig.1 shows the as-cast microstructure of alloys. Fig.1 (a) is amplified 50 times, and Fig.1 (b) is amplified 500 times. It can be seen the microstructure appears daisy-like, cast alloy is mainly consist of α--Mg base solid solution (B) and eutectic phase Mg_5RE, $Mg_{24}RE_5$. Eutectic phase is mainly distributed on the grain boundary as continuous mesh (A), which shows block or sliver irregularly shaped. The intracrystalline precipitate is less, which dispersion and distribution, mainly shows point and ellipsoidal (C) forms. Fig.2, Fig. 3 and Fig.4 showed the EDS spectrums quantitative analysis data for different point.

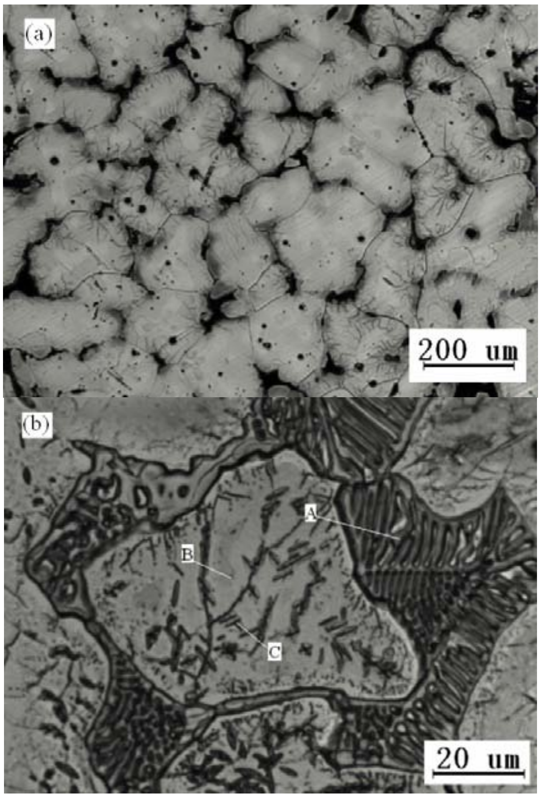
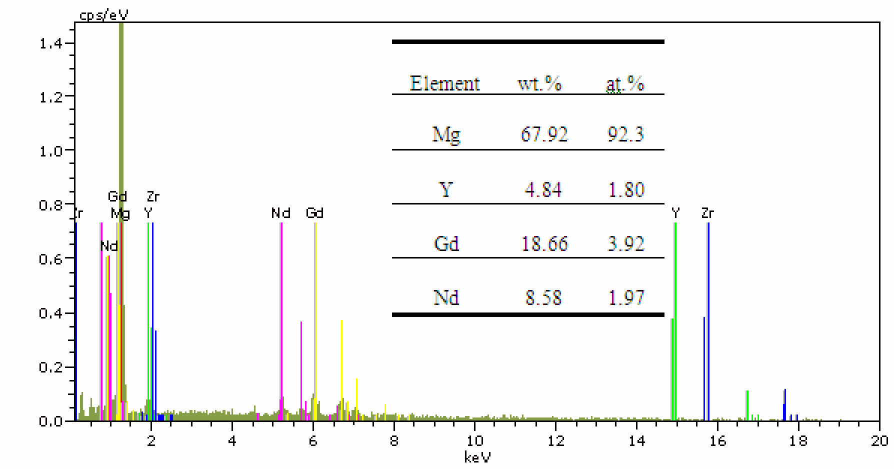
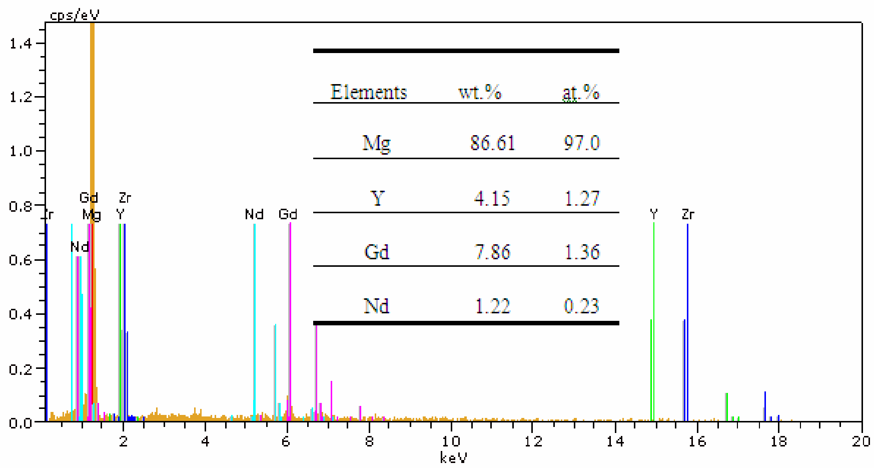


Fig.1. As-cast microstructure (a) 50X (b) 500X.



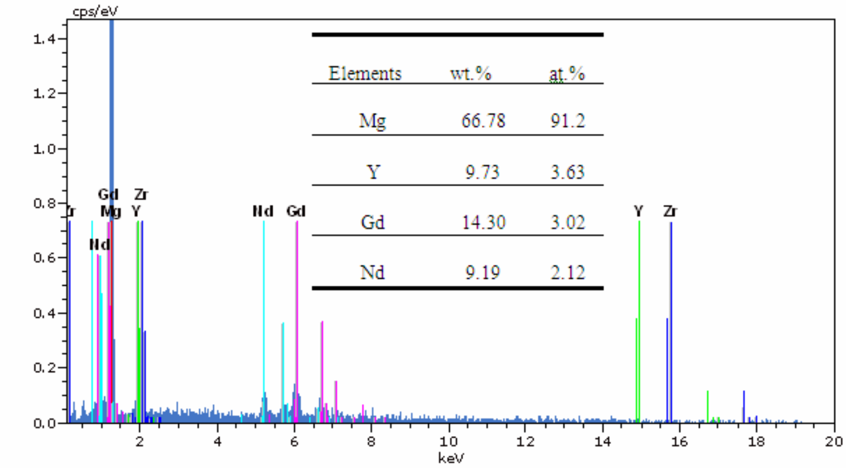
Element	wt.%	at.%
Mg	67.92	92.3
Y	4.84	1.80
Gd	18.66	3.92
Nd	8.58	1.97

Fig. 2. EDS analysis for B point.



Elements	wt.%	at.%
Mg	86.61	97.0
Y	4.15	1.27
Gd	7.86	1.36
Nd	1.22	0.23

Fig. 3. EDS analysis for A point .



Elements	wt.%	at.%
Mg	66.78	91.2
Y	9.73	3.63
Gd	14.30	3.02
Nd	9.19	2.12

Fig.4. EDS analysis for C point.

EDS in SEM analysis reveals the phase of B point is magnesium based solid solution containing Y, Nd and Gd, A and C point are Dendrite-like and ellipsoid-shaped eutectic phase respectively. The total content of rare earth elements on the grain boundary is more than in matrix, average grain size of as-cast alloys is about 105 μm.

Fig. 5 showed optical microstructure of the deformed alloy by two pass. It could be seen the black network eutectic were broken into many tiny grains on the grain boundary, diffusion distributed on the grain boundary after deformation.

Obviously grain refinement by dynamic recrystallization appeared in local regional of grain boundaries (Fig. 5 (a)). All the grains were stretched along with deformation direction, grain size were obviously refined, about 86 μm, but its distribution is non-uniform. Stacking fault energy of magnesium alloy is low, instead of dynamic recovery, dynamic recrystallization easy occurred in hot processing. The fine eutectic phase hindered the grow of α-Mg solid solution, play the role of pinning grain boundaries and refined the microstructure of the magnesium alloy. Dynamic recrystallization took place in the specimen and caused softening, this also caused a transition from brittle to ductile behavior, which leads to improve the ductility [10]. The most fundamental reason the plasticity of the magnesium alloy can be greatly improved is non-basal slip is activated. Research has shown that non-basal slips are prone to occurrence near the grain boundary. Grain refinement can activated potential non-base slip system such as prism surface and taper surface slip in the magnesium alloy.

Simultaneously, from the amplified microstructure, fine precipitates phases with the point shape also scattered inside the matrix can be observed (Fig. 5 (b)). It suggests that dynamic precipitation occurred during deformation. Because of driving force of dynamic precipitation and dynamic recrystallization comes from the deformation, so the dynamic precipitation will reduced driving force of recrystallization nucleation, and will prevent occurring dynamic recrystallization. As well we know, for magnesium alloy, when deformation temperature increasing or the grain refinement, the non-basal slip will be activated. Grain boundary sliding and potential non-base slip systems (pyramid slip and cone slip) were started by heat activation, so the plasticity of magnesium alloy has been greatly improved. In this experiment, these precipitations have pinning effect on the grain boundary migration, which prevent the geometric dynamic recrystallization. Therefore large amounts of dynamic precipitation eventually inhibit occurring dynamic recrystallization in alloy.

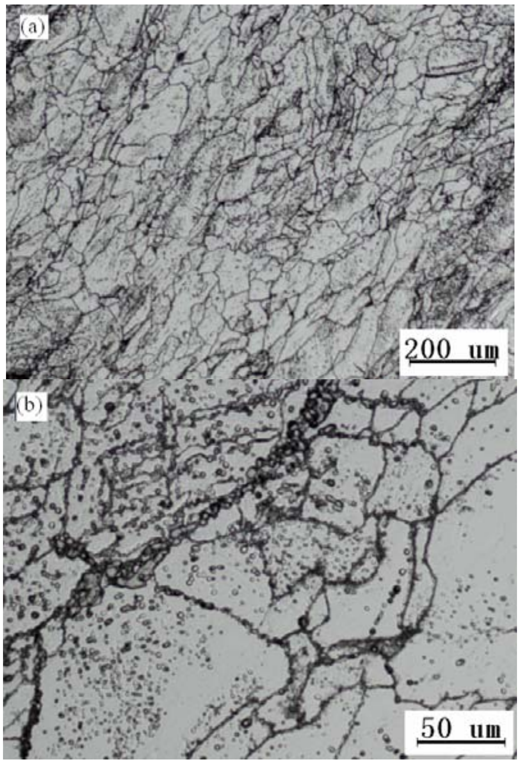


Fig. 5. Deformed microstructure (a) 50X (b) 500X.

3.2 Comparison of the mechanical properties between T5 and T6 condition

As can be seen from Tab. II and Tab.III, the tensile strength of magnesium alloys after T6 heat treatment is 242Mpa, the elongation is 5.17%. Compared with T6 heat treatment, large improvements of ultimate tensile strength (UTS) are observed after T5 heat treatment. Its tensile strength increased to 359.3Mpa, increased by approximately 48.5%; Elongation is increased from 5.17% to 6.5%, increased by approximately 25.7%. It showed the comprehensive properties are obviously improved from T6 to T5 heat treatment.

Tab.II Mechanical properties of specimen after T6 heat treatment.

No.	Ultimate tensile Strength /MPa	Elongation /%
1	Rm=244	A=5.5
2	Rm=242	A=4.5
3	Rm=240	A=5.5
Average	Rm=242	A=5.17

Tab. III Mechanical properties of specimen after T5 heat treatment.

No.	Ultimate tensile Strength /MPa	Elongation /%
1	Rm=362	A=6.0
2	Rm=356	A=7.0
3	Rm=360	A=6.5
Average	Rm=359.3	A=6.5

3.3 Microstructure after heat treatment

From the fig.6, Dendrite structure of magnesium alloys has disappeared after solution treatment. Rich rare phase on the grain boundary has been largely dissolved into the matrix, and grain boundaries have become clear, all the grains become regular equiaxial grains.

During the solid solution treatment, grain-boundary diffusion and migration ability are increased. Dislocations were cancelled each other and nuclear of recrystallization were reduction. This led to grain coarsening, parts of grains have grown up after solid solution treatment, and grain size is about 64μm on average. After ageing followed solution heat treatment (Fig.7), grain size has decreased and several grey-black second phases were precipitated and scattered in the grain. Distribution of grain is more even than the solution, but grain size is still non-uniform.

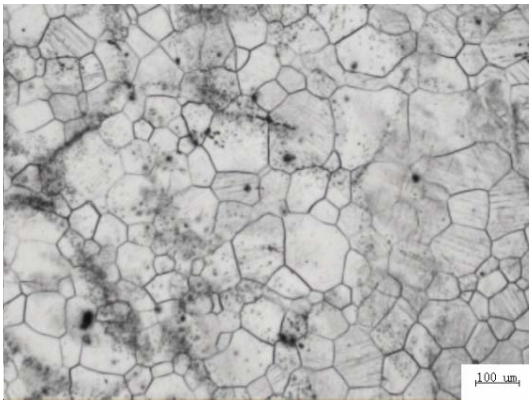


Fig. 6. Microstructure after solution treatment.

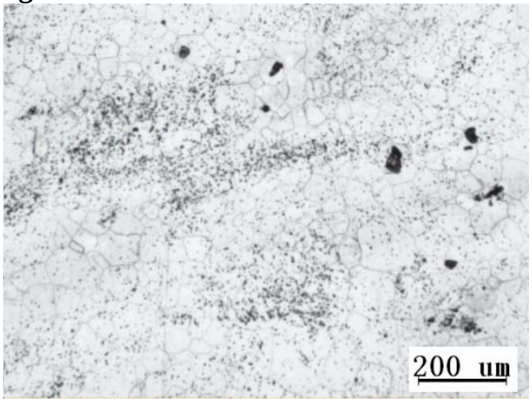


Fig. 7. Microstructure after aging following solution treatment.

Fig. 8. is the microstructure after direct ageing treatment. As can be seen from the microstructure after T5 treatment, the grains were much smaller than T6 condition, the grain

size was less than 5μm. The second phase on the grain boundary were refined and more evenly dispersion.

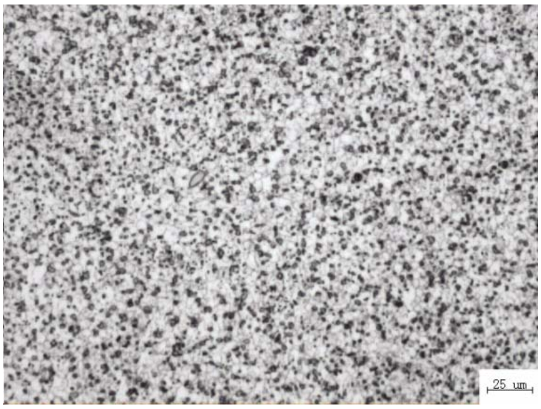


Fig. 8. Microstructure of T5 condition.

According to Hall-Patch equation: $y = \sigma_0 + kd^{-1/2}$, the yield strength of alloy is inversely proportional to the square root of the grain size. Studies have shown that material strength is increased with refining grain size, at the same time plasticity is improved noticeably. Compared with body-centered cubic and face-centered cubic crystal, refine strengthening is more effective to improve the mechanical properties of magnesium alloy. This is the main reason that T5 condition has higher mechanical properties than the T6 condition. On the other side, the remarkable age-hardening response achieved in the Mg-Gd-Y alloys is attributed to a dense distribution of precipitates in the microstructure. Except that fine-grain strengthening, ageing precipitated phase can also impede dislocation motion, which can increase the yield strength of the material. Ageing precipitation sequence of this alloy is complex and metasTab. transition phase is much more. Apps and others [11-13] also confirmed the presence of the β1-phase in Mg-7Gd-2.25Nd (wt.%) Alloys, believed the ageing sequence is: α-Mg(ssss)→β''(DO19)→β'(BCO)→β_1(FCC)→β(Mg_5Gd, FCC□,this is resemble as Mg–Gd alloys. For the Mg–Gd alloys, the precipitation involves forms β'', β', β_1 and β[14,15].β' phase have a base-centred orthorhombic structure (a=0.64nm, b=2.2nm, c=0.52nm) the orientation relationship is (100) β'//(110)α and $[001]_{\beta1}//[0001]_{\alpha}$.The β_1 phase has a face-centered cubic structure (FCC,a=0.74 nm), and the orientation relationship is(112)//$(100)_{\alpha}$ and $[001]_{\beta1}//[0001]_{\alpha}$. β_1 and β phase will make alloy occur over ageing, which weaken the reinforcement of alloy.

Research believe the generation of β' phase is mainly reason for improvement of alloy's mechanical properties, β' phase and the phase of the matrix is semi-coherent interface, matching is relatively high. The grain boundary precipitated phases have obvious pinning effect to the alloy's grain boundary. It prevents the grain growth by ageing for a long -time. Strengthening role of β' precipitation phase make its strength increased significantly.

4. Conclusions

[1] Dynamic precipitation and dynamic recrystallization appeared in the microstructure at the same time after deformation. Dynamic precipitation will reduce the driving force of recrystallization nucleation and inhibiting the dynamic recrystallization.

[2] After T5 treated, the comprehensive mechanical properties of deformed alloy improved larger than T6 condition. The tensile strength is 359.3Mpa, increased by approximately 48.5%; Elongation is 6.5%, increased by approximately 25.7%.

[3] Grains growing up significantly after T6 treatment, it will affect the strength of the alloy. After T5 treatment, precipitated phase have obvious pinning effect to grain boundary

and prevent the grain growth ageing for a long-time. It can effectively improve the comprehensive mechanical properties of alloy.

Acknowledgements

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5. References

1. S.Ristic, A.Jovicevic, S.Kocic, M.Spasojevic. Development of New Material for the Environmental-Friendly Energy Generation Processes. Science of Sintering, 43 (2011) 71-79.
2. Rokhlin L L. Magnesium Alloys Containing Rare Earth Metals[M].London: Taylor and Francis,2003.
3. Lorimer GW. In: Baker C, Lorimer GW, Unsworth W, editors. Proceedings of the London conference on magnesium technology. London: The Institute of Metals; 1987:47–53.
4. S. Kamado, Y. Kojima, R. Ninomiya, K. Kubota, in: G.W. Lorimer (Ed.),Proceedings of the Third International Magnesium Conference, 1996, Institute of Materials, Manchester, UK, 1997:327.
5. Rokhlin L L,Nikitina N I. Magnesium-gadolinium and magnesium-gadolinium-yttrium alloys[J].Z Metallkd,1994,85: 819.
6. Kamado S,et al. Aging hardening characteristics and high temperature strength of Mg-Gd and Mg-Tb alloys[J]. Jpn Inst Light Met,1992,42(12):727.
7. Y. J. Wu, Z. M. Zhang, B.C. Li. Effect of Aging on the Microstructures and Mechanical Properties of AZ80 and ZK60 Wrought Magnesium Alloys. Science of Sintering, 42 (2010) 161-168.
8. Kawabata T□Matsuda K□Kamado S,et al□HRTEM observation of the precipitates in Mg-Gd-Y-Zr alloy[J]. Mater Sci Forum,2003,303:419.
9. Anyanwu LA, Kamado S, Kojima Y. Aging characteriatics and high temperature tensile properties of Mg-Gd-Y-Zr alloys[J]. Materials Transactions,2001,42(7):1206.
10. Socjusz-Podosek M, Lityneska L. Effect of yttrium on structure and mechanical properties of Mg alloys[J]. Mater Chem Phys,2003,80(2):472.
11. T.Al-Samman,G.Gottstein. Dynamic recrystallization during high temperature deformation of magnesium. Materials Science and Engineering A 490(2008)411-420.
12. Apps P J, Karimzadeh H, King J F, Lorimer G W. Phase compositions in magnesium-rare earth alloys containing yttrium, gadolinium or dysprosium [J]. Scripta Materialia, 2003,48:475–481.
13. Nie J F, Muddle B C. Characterizations of strengthening precipitate phases in a Mg-Y-Nd alloy[J]. Acta Materialia 2000, 48:1691–1703.
14. Apps P J, Karimzadeh H, King J F, Lorimer G W. Precipitation reactions in Magnesium-rare earth alloys containing Yttrium, Gadolinium or Dysprosium [J]. Scripta Materialia, 2003, 48: 1023–1028.
15. Jian feng NIE. Precipitation and Hardening in Magnesium Alloys[J]. Metallurgical and Materials transactions A. Volume 43A, November 2012:3891-3939.

Садржај: *У раду је проучавана еволуција микроструктуре Mg - Gd - Y - Nd - Zr легуре Mg отпорне на топлоту након деформације и третмана T5 или T6. При термопластичној деформацији, у исто време се дешавају и динамичка рекристализација и динамичка преципитација. Динамичка преципитација редукује покретачку силу нуклеације; што даље спречава појаву динамичке рекристализације. Чвсти раствор настаје на 530 ^oC, 4h. Третмани старења се одигравају на 225 ^oC током 16h. Легура је показала боља свеобухватна својства током T5 у односу на T6 третман. Након T5 третмана, чврстоћа је порасла до 359.3 МПа, око 48.5%; елонгација је порасла од 5.17% до 6.5%. Након третмана старења, β фаза је главна. . У исто време, улога преципитационе фазе је кључна и чини материјал значајно чвршћим.*

Кључне речи: *еволуција микроструктуре, Mg-Gd-Y-Nd-Zr, легура магнезијума, чврсти раствор, третман старења*