Deformation behaviour and microstructure development of Twin Roll Cast AZ31 strips

M. Ullmann, R. Kawalla, H.-P. Vogt

The new energy efficient method of producing magnesium strips down to 1,0 mm thickness based on Twin Roll Casting (TRC) and Strip Rolling in industrial scale developed at the Institute of Metal Forming at the TU Bergakademie Freiberg in cooperation with the MgF Magnesium Flachprodukte GmbH Freiberg (Germany) is described. The design of rolling schedule was analyzed, based on the control of the recrystallization behaviour, to achieve fine grain in the strip rolling process of the magnesium alloy AZ31. The dynamic recrystallization behaviour of this alloy during hot deformation was determined with help of plane strain compression tests and microscopic examination. The influence of temperature, strain and strain rate on the activation of dynamic recrystallization process during deformation was analyzed. The deformation behaviour was also simulated with semi-empirical models including specific coefficients related to processing parameters (strain, strain rate and temperature). The results show that the grain size of this alloy refine steadily with increasing rolling passes by dynamic recrystallization. The tensile strength and ductility were improved correspondingly.

Keywords: Twin roll casting - Strip rolling - AZ31 - Magnesium alloy - Dynamic recrystallization - Plain strain compression test - Flow curve

INTRODUCTION

As lightest metallic construction material with a density of 1,74 g/cm³, magnesium is attractive for many modern construction concepts and engineering solutions for its properties like high electro-magnetic damping, good weight-strength ratio and a high recycling capability. However, it HCP lattice structure requires a processing temperature of 225 °C or above to obtain the desired forming abilities. This feature makes the material vulnerable at high deformation rates. Therefore the conventional hot rolling chain of slab must consist several intermediate annealing steps to overcome this shortage, which increases the time and energy consume. This complex production route makes magnesium sheet at the weaker position in the competing market with aluminium alloy.
A new production technology combined Twin Roll Casting and subsequent rolling for thin sheet-material is investigated at the TU Bergakademie Freiberg for solving this problem and making Mg-sheets a more economical material for wide-range application in automotive and other mobility industries.

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Twin Roll Casting and Strip Rolling in Freiberg

The processing route developed at the Institute of Metal Forming (IMF) consists of a primary Twin Roll-Casting (TRC) step, followed by a homogenization heat treatment and a subsequent rolling to the desired final thickness. This technology allows the production of near-net-shape Mg-sheets in the primary production step, making several of the rolling and annealing steps of the conventional technology negligible. Instead, the final thickness for the sheet products can be reached by 2-4 rolling passes, making this kind of processing faster and cheaper by saving time and energy.
For the TRC-process, the melt is cast by a special nozzle between two horizontal, water-cooled working rolls. Due to the contact with the cooled rolls, a rapid solidification takes place and so a primary deformation is introduced to the material until leaving of the roll gap. Prior to coiling the edges of the TRC-material are trimmed and the TRC-coil is then cooled down to room temperature. Afterwards the material is homogenized and heated to the desired rolling temperature and rolled down to the desired final thickness.
The whole processing route is shown schematically in Fig. 1. For realization of this production line, the IMF is

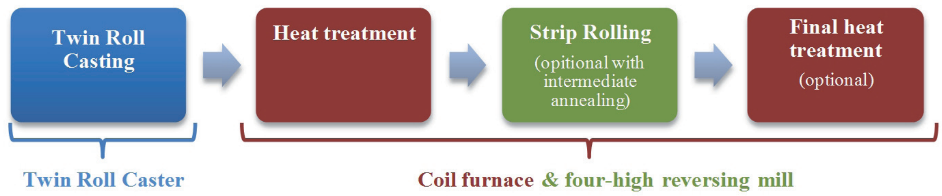


Fig. 1 - Schematic diagram for the TRC- and hot rolling process for magnesium sheet at the Institute of Metal Forming.

Fig. 1 - Diagramma schematico del processo TRC – e di laminazione a caldo di lamiere di magnesio presso l'Institute of Metal Forming.

equipped with a prototype Twin Roll Caster, a coil heating and annealing furnace and a four high quarto reversing mill (Fig. 2). A summary of the technical data for both prototype plants is given in Tab. 1.

	Twin Roll-Caster	Four high reversing mill
max. roll force	7 MN	12 MN
max. roll torque	200 kNm	130 kNm
max. strip speed	3 m/min	225 m/min
max. strip width	780 mm	720 mm
final strip thickness	3 – 7 mm	≥ 1.0 mm
work roll diameter	840 mm	400 mm

Tab. 1 - Technical data of the Twin Roll Caster and the four high reversing mill for production of Mg-strip at the Institute of Metal Forming.

Tab. 1 – Dati tecnici dell'impianto TRC e della gabbia quarto reversibile per la produzione di nastri di magnesio presso l' Institute of Metal Forming.

These two pilot-research plants are designed for the investigation and development of TRC- and strip rolling technology for magnesium alloys in industrial scale. Therefore the melting furnace is also utilized for the use of recycling material and the rolling mill is equipped with industrial working features like a flatness measuring system, minimum quantity lubrication and a coiling system for the application of strip tension (usually 20-50 kN). Additionally the TRC-plant can be equipped with a smaller prototype TRC-system with a melt capacity of 250 kg for production of smaller strip with approx. 300 mm in width for development of a TRC-technology for new or enhanced magnesium alloys.

With the current technical development, the aim is to develop a rolling technology for the production of magnesium strips with a minimal final thickness of 1,0 mm and improved properties under stable process conditions. Currently, both process stages, Twin Roll Casting and strip rolling, have been tested for the magnesium alloys AZ21, AZ31, AM20, AM40, AM50, ZE10, ME21 and WE43.

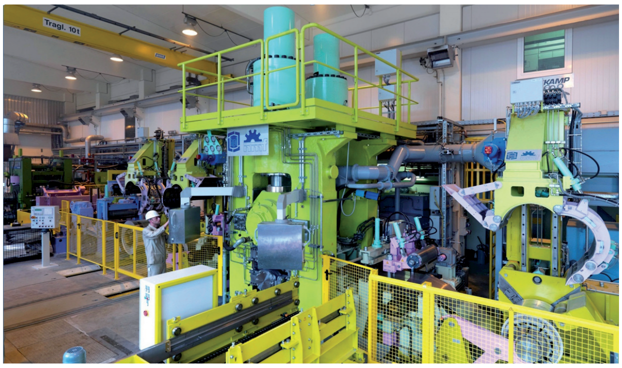


Fig. 2 - TRC- and Strip Rolling process for magnesium strip at the Institute of Metal Forming.

Fig. 2 - Processo TRC – e laminazione di nastri di magnesio presso l' Institute of Metal Forming.

STRIP ROLLING TESTS WITH TRC MATERIAL AND THEIR RESULTS

Main emphasis in research activities is the development of a TRC-technology for the widely used and well known AZ31 Mg-alloy. During TRC of magnesium alloys containing aluminium and zinc, the formation of segregations and brittle precipitates cannot be completely suppressed. Phases, such as Mg-Al-Zn, exist in interdendritic interstices. Two types of second phases particle can be observed: Mg-Al-Zn ($(Al,Zn)_{49}Mg_{32}$ [1] or $Mg_{17}Al_{12}$ phase [2]) and Al-Mn (that could be the Al_8Mn_5 phase [2] or a mixture of $Al_{11}Mn_4$, Al_8Mn_5, Al_9Mn_{11} phases [3]). In order to produce a homogeneous initial structure, a suitable heat treatment is required to dissolve these phases or to distribute them. The initial dendritic microstructure after TRC is converted into a bi-modal grain structure after the homogenization annealing (Fig. 3) with a mean grain size in the range of 20-25 µm. During TRC forming energy is introduced locally into the material and causes static recrystallization in these areas. In the following rolling step, the primary grains are stretched, and leading to dynamic recrystallization, which is normally inhomogeneous (Fig. 4). The recrystallization starts in the most deformed areas. Dislocations pile up at the grain boundaries, the grain boundary areas therefore

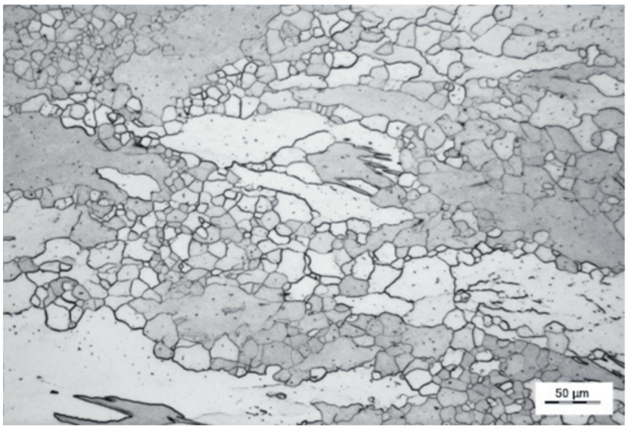


Fig. 3 - Microstructure of AZ31 after TRC-process and homogenization.

Fig. 3 – Microstruttura della lega AZ31 dopo processo TRC e omogeneizzazione.

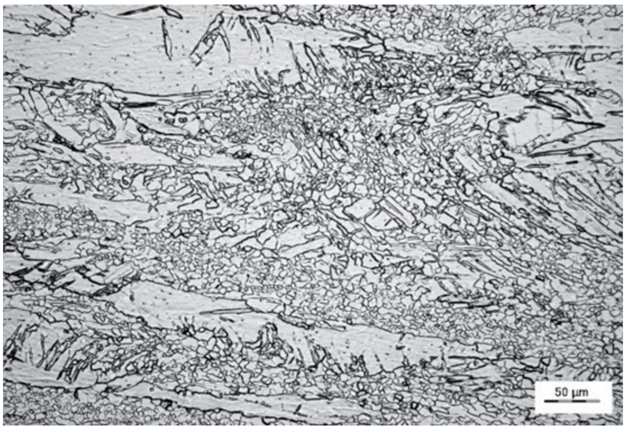


Fig. 4 - Microstructure after TRC and rough rolling.

Fig. 4 – Microstruttura dopo processo TRC e sbozzatura.

Fig. 5 - Microstructure after TRC, rough rolling and intermediate annealing.

Fig. 5 – Microstruttura dopo processo TRC, sbozzatura e ricottura intermedia.

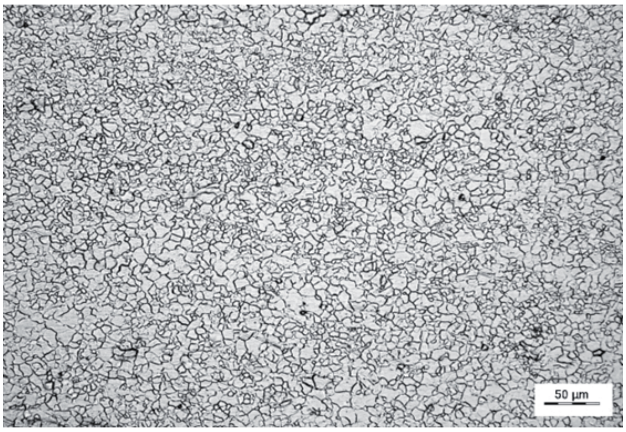
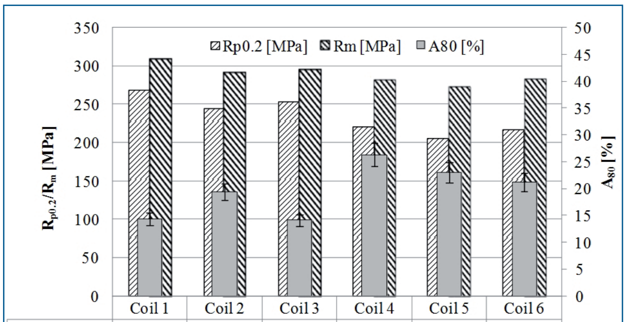


Fig. 6 - Microstructure after intermediate annealing and finish rolling.

Fig. 6 – Microstruttura dopo ricottura intermedia e laminazione di finitura.

serve as nucleation sites of recrystallization. Furthermore, the contained fine precipitates (particles) increase the stored energy and thus the driving force for recrystallization. The large incoherent particles act as nucleation sites, while the finely dispersed precipitates pinning the grain boundaries [4]. In areas where the available deformation and temperature is too low for a complete recrystallization, twins are formed.
After intermediate annealing the bimodal structure is almost removed by static recrystallization processes (Fig. 5). An intermediate heating of the strip after the second rolling pass triggers static recrystallization and marginal grain growth. During the subsequent rolling passes, which are characterized by a controlled setting of a sufficient temperature, rolling speed and reduction per pass, a further grain refinement by dynamic recrystallization takes place. The average grain size of the finished strip is approximately 5 µm (Fig. 6). Due to the intermediate heating, the strip characteristics in width and length tend to equalize already in the rolled condition. In addition, a stable process control is achieved as the strip edges are less prone to cracking.
Strip rolling tests without intermediate heating after the 2nd pass have shown that fully recrystallized grains can only be achieved at a very high deformation. In partially recrystallized microstructure, twins and stretched grains form in addition to shear bands or “shear band - like” structures [5]. Consequently, the microstructure of the finished strip is inhomogeneous [see also [6]). As already described above, the intermediate annealing leads to a more uniform and finer microstructure and therefore to higher elongation values then after rolling without intermediate heating can be achieved (Fig. 5 and 6).
The development of the mechanical properties in the strip is shown in Fig. 7. The initial TRC-state shows a combination of high strength with very poor formability. After

the homogenization heat treatment, the deformation to fracture is strongly enhanced with the yield strength being lowered. Due to the fine grained, homogeneous structure after rolling, the final strip shows a combination of high strength up to 300 MPa and high failure strain up to 25 %. However, strips rolled without intermediate heating can also reach elongation of 25 ± 2.5%, with a slight loss in strength applying an appropriate final annealing.
Next to AZ31 a wide range of different magnesium alloys have been Twin Roll Cast and investigated until now, including quite common alloys as the AM-series (AM20, AM40, AM50) and also including rare-earth alloys as MnE21 and WE43. The TRC-processing route is a fast and effective way to obtain thin sheet and strip of magnesium alloys with good and homogeneous properties, offering the option to produce semi-finished products and final products with reduced costs.



	Coil 1	Coil 2	Coil 3	Coil 4	Coil 5	Coil 6
Rp0.2 [MPa]	269	245	254	221	206	217
Rm [MPa]	309	291	294	280	271	281
A80 [%]	14,3	19,4	14,1	26,3	23	21,2

Fig. 7 - Influence of intermediate annealing on mechanical properties during rolling of 1,5 mm thick AZ31 strips in four passes.

Fig. 7 – Influenza della ricottura intermedia sulle proprietà meccaniche durante la laminazione in quattro passaggi di nastri di lega di magnesio AZ31 con spessore di 1,5 mm.

MODELING THE SOFTENING BEHAVIOUR

To use TRC magnesium strip in the following rolling process, it is necessary to specify the requirements for the property profile of the TRC strip and also the basic knowledge of plasticity and deformation behaviour of the metal. In this way it is possible to optimize both properties, as well as technologies to encourage the application of wrought magnesium alloys. This includes the information concerning the hardening and softening behaviour in temperature regions suitable for the used magnesium alloys, and also the knowledge concerning the correlation between microstructure and properties. Simultaneously, these factors are also the basic requirement for a numeric simulation of the strip rolling process.

Feedstock and experimental procedure

The feedstock for the rolling samples was Twin Roll Cast Magnesium strips (width 700 mm, thickness 5,2 mm, alloy AZ31), produced on a pilot plant originating from a cooperation between the TU Bergakademie Freiberg and the MgF Magnesium Flachprodukte GmbH. The chemical composition of the samples taken from the material is as follows: Mg 96,2 %; Al 2,7 %; Zn 0,7 %; Mn 0,36 %; residual 0,04 %. At the beginning all samples were annealed at 430 °C for six hours in order to assure a homogeneous initial state. Subsequently the samples were brought to deformation temperature and were tested with help of plain strain compression tests. Two different methods were used: continuous plain strain compression tests for characterizing dynamic softening and discontinuous ones (using the offset-method) for the assessment of static recrystallization. The experimental investigations included tests in the temperature range between 250 and 400 °C and strain rates between 0,1 and 10^{s-1}. The tests were evaluated concerning flow stress, recrystallization kinetics and grain growth.

Model approaches and results

In order to model a dynamical microstructure development, physical-empiric approaches are used whereas process conditions will be correlated to the characteristics of the material. The coupled influence of strain rate and thermal activation of hot forming is described with the help of the Zener-Hollomon parameter Z (see Tab. 2) [7]. After exceeding the critical deformation degree the dynamic recrystallization of the material starts. The dynamically recrystallized fraction of microstructure X_{dyn} is described by equation 4 in Tab. 2. The mathematical description of the recrystallized grain size D_{dyn} follows equation 5 which delivers in combination with equation 2 the correlation between flow stress level and dynamically recrystallized grain size [8; 11]. A parameter fit is performed according to the experimental results of the material's flow behaviour. Thereby, the run of the flow curve reveals information on the particular softening mechanism [9; 12], see also Fig. 8, left. If dynamic recrystallization takes place incompletely or a forming condition of $\varphi < \varphi_c$ exists, the opportunity of a static recrystallization is provided in pause times (see Tab. 3) [10]. The statically recrystallized fraction of microstructure is determined depending on forming conditions and temperature using a JMAK approach. Besides the grain size, forming temperature and the comparative deformation degree as well as a reprehensive strain rate of the last forming step are the main influencing parameters on the statically recrystallized share of microstructure and its grain size.
The flow curves (exemplarily shown in Fig. 8, top right) show that TRC AZ31 softens preferably by dynamic recrystallization during hot deformation. Even at low deformation temperature of 250 °C, dynamic recrystallization begins above critical deformation degree of 0,2. At a high

Grain growth		$D_{KW}^{a} = D_{0}^{a} + A \cdot t \cdot \exp\left(-Q_{KW} / (R \cdot T)\right)$	
Zener-Hollomon parameter		$Z = \dot{\varphi} \cdot \exp\left(\frac{Q_w}{R \cdot T}\right) = A \cdot [\sinh(\alpha \cdot \sigma_{\max})]^m$	
Strain at which the dynamic recrystallization begins		$\varphi_c = a_1 \cdot D_0^{a_2} \cdot Z^{a_3}$	
Fraction at static recrystallization		$X_{dyn} = 1 - \exp\left[e_1 \cdot \left(\frac{\varphi - \varphi_c}{\varphi_{0,5} - \varphi_c}\right)^{e_2}\right]$ $\varphi_{0,5} = c_1 \cdot D_0^{c_2} \cdot \exp\left(\frac{c_3}{T}\right) \cdot \dot{\varphi}^{c_4}$	
Grain size at dynamic recrystallization		$D_{dyn} = d_1 \cdot Z^{d_2}$	
φ	equivalent plastic strain	D_{dyn}	dynamically recrystallized grain size
$\dot{\varphi}$	equivalent deformation rate	D_{KW} Q_w	grain size due to grain growth activation energy in the dynamic model
T	temperature	A,α,m	parameters to determine Z
σ_{max}	Maximum yield stress	d_1,d_2	parameters to determine D_{dyn}
Z	ZENER-HOLLOMON parameter	a_1-a_3	parameters to determine φ_c
φ_c	strain, at which the dynamic recrystallization starts	e_1,e_2	parameters to determine X_{dyn}.
φ_{05}	strain, at which 50% of the microstructure is dynamically recrystallized	c_1-c_4	parameters to determine φ_{05}
X_{dyn}	dynamically recrystallized volume fraction	A,a	parameters to determine the grain growth

Tab. 2 - *Models quantifying the dynamic recrystallization.*

Tab. 2 - Parametri che quantificano la ricristallizzazione dinamica.

Fraction at static recrystallization		$X_{stat} = \left(1 - \exp\left[-h_1\left(\frac{t - t_0}{t_{0,5}}\right)^{h_2}\right]\right)$ $t_{0,5} = g_1 \cdot \varphi^{g_2} \cdot D_0^{g_3} \cdot \dot{\varphi}^{g_4} \cdot \exp\left(\frac{Q_{stat}}{R \cdot T}\right)$	
Grain size at static recrystallization		$D_{stat} = s_1 \cdot \varphi^{-s_2} \cdot D_0^{s_3} \cdot Z^{-s_4} + s_5$	
Grain growth		$D_{KW}^{a} = D_{0}^{a} + A \cdot t \cdot \exp\left(-Q_{KW} / (R \cdot T)\right)$	
$t_{0,5}$	time, at which 50% of the microstructure is statically recrystallized	t_0	time, at which static recrystallization start
g_1-g_4	parameters to determine $t_{0,5}$	h_1,h_2	parameters of AVRAMI-kinetic
D_{stat}	statically recrystallized grain size	D_0	initial austenite grain size

Tab. 3 - Models quantifying the static recrystallization.

Tab. 3 - Parametri che quantificano la ricristallizzazione statica.

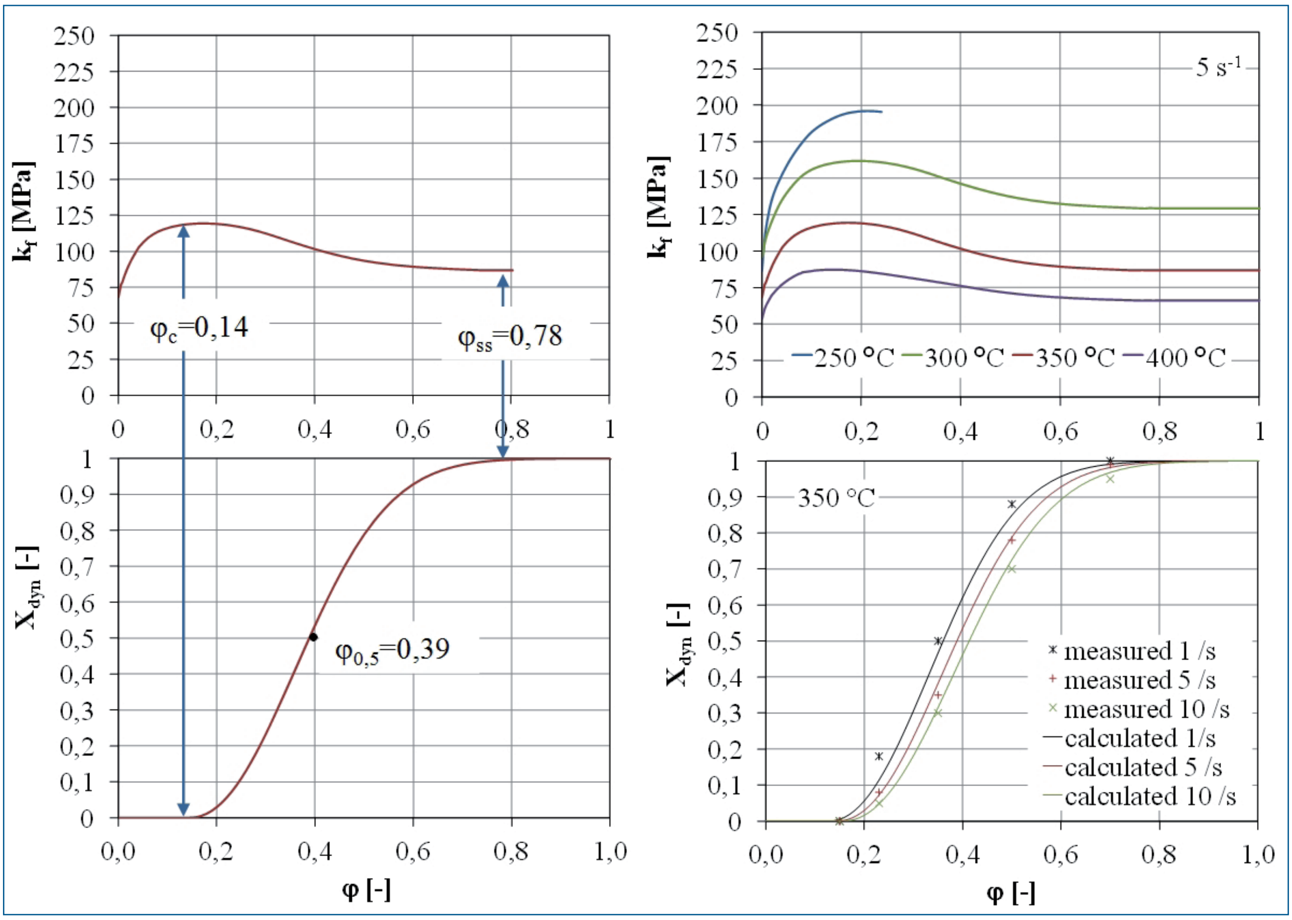


Fig. 8 - Dynamic recrystallization based on the flow curve (left) determined at 350 °C, 5 $^{s-1}$ and flow curves depending on temperature, strain rate 5 $^{s-1}$ (top right) as well as recrystallized fraction depending on strain rate, 350 °C (down right).

Fig. 8 – Ricristallizzazione dinamica sulla base delle curve di flusso (a sinistra) determinata a 350 °C, 5 $^{s-1}$ e curve di flusso dipendenti da temperatura, velocità di deformazione 5 $^{s-1}$ (in altro a destra) e frazione ricristallizzata in funzione della velocità di deformazione a 350 °C (in basso a destra).

deformation temperature of 400 °C, dynamic recrystallization starts already at a deformation degree of 0,11 (there is only a low dependency on strain rate). An almost completely recrystallized structure exists at this temperatures at strains of approximately 0,7. Fig. 8, down right, presents an example at temperature of 350 °C and different strain rates (1, 5 and 10 $^{s-1}$) of the correlation between deformation degree and dynamic fraction of recrystallization.
Fig. 9 and 10 illustrate an example of the influence of temperature on the recrystallization kinetics. At low temperatures a higher degree of deformation has to be applied due to the relatively low thermal activation for a complete dynamic recrystallization. At low temperatures the dissipation energy must compensate the lack of thermal activation, whereas at high temperatures already a very low degree of deformation is sufficient for recrystallization. Fig. 11 illustrates the dependency of the dynamic grain size on Zener-Hollomon parameter, which indicates the effect of temperature and strain rate. The mean grain size of dynamic recrystallization was found below 10 µm for the investigated samples.

The dynamic and static recrystallized shares were respectively obtained from the test results and characterized. They show the typical course after JMAK theory. The dynamically and statically recrystallized grain sizes are largely determined by the initial grain size, the temperature, the degree of deformation and strain rate, as well as by the pause time for static softening. It was a mathematical description of these sizes on hand of JMAK theories. The magnesium alloy AZ31 softens dynamically preferably at temperatures above 250 °C.

SUMMARY AND FUTURE PROSPECTS

Magnesium Twin Roll Casting is going to emerge as the future producing method for magnesium strips as a result of its economic and material-specific advantages.
The most important goal during strip rolling process is to achieve a suitable final product for different applications. After strip rolling the microstructure is homogeneous. The average grain size of the final product is approximate-

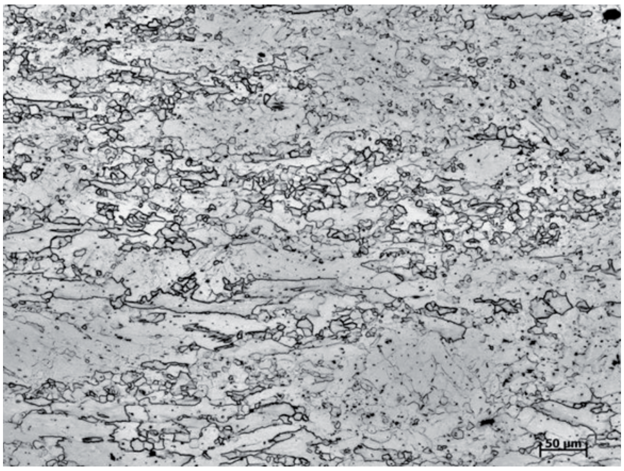


Fig. 9 - Partially recrystallized microstructure (32 % recrystallized fraction, 200-fold magnification), ϑ = 300 °C; φ_V = 0,35; ϕ = 5 s^{-1}.

Fig. 9 - Microstruttura parzialmente ricristallizzata (32 % di frazione ricristallizzata, ingrandimento 200x), ϑ = 300 °C; φ_V = 0,35; ϕ = 5 s^{-1}.

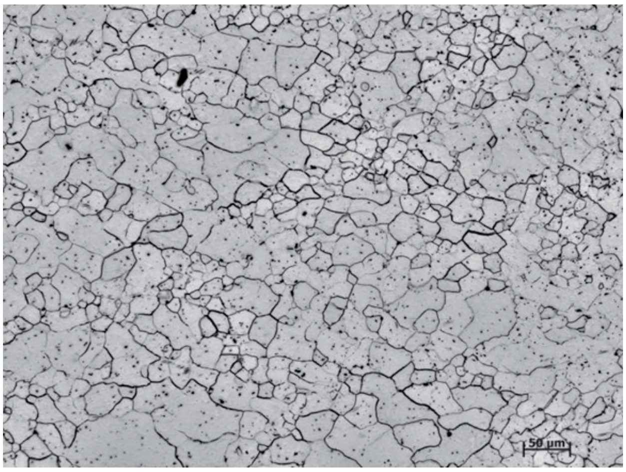


Fig. 10 - Completely recrystallized micro-structure (100 % recrystallized fraction, 200-fold magnification), ϑ = 400 °C; φ_V = 0,7; ϕ = 5 s^{-1}.

Fig. 10 - Microstruttura completamente ricristallizzata (100 % di frazione ricristallizzata, ingrandimento 200x), ϑ = 400 °C; φ_V = 0,7; ϕ = 5 s^{-1}.

ly 5 µm. Due to which yield points of 220 MPa, tensile strengths of 280 MPa and total elongations of 25 ± 2,5% are reached for AZ31 strips.
It was found, that this TRC AZ31 softens preferably by dynamic recrystallization. For technical processes the temperature range above 250 °C is interesting. Dynamically recrystallized grain sizes below 5 µm are reachable by adjusting Zener-Hollomon Parameter.
Currently the material coefficients for the models of the two different initial microstructures – the TRC and the continuous cast and rolled one – are compared.
In future work the models should be implemented in a numeric simulation of the strip rolling process for optimizing the process parameters. Besides the model approaches should be expanded to other magnesium alloys, e.g. the AM alloys.

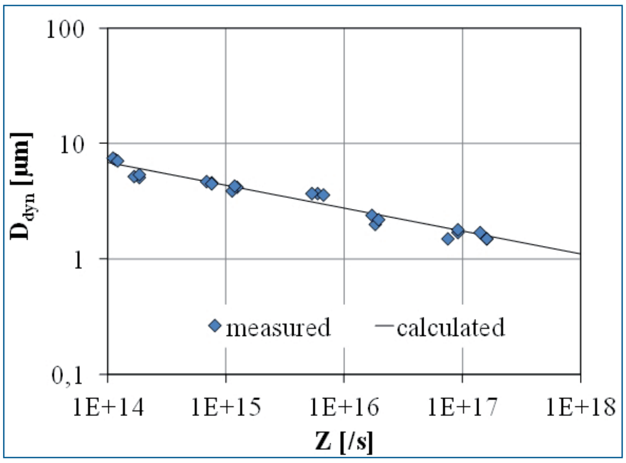


Fig. 11 - Dynamic grain size depending on Zener-Hollomon parameter.

Fig. 11 – Dimensione dinamica del grano in funzione dei parametri Zener-Hollomon.

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REFERENCES

1] G.F. Vander Voort; Metallography and Microstructures, Vol. 9, ASM International, 2004
2] T. Laser, M.R. Nürnberg, A. Janz, Ch. Hartig, D. Letzig, R. Schmidt-Fetzer, R. Bormann; The influence of manganese on the microstructure and mechanical properties of AZ31 gravity die cast alloys; Acta Materialia 54, 2006, pp. 3033-3041
3] P. Cao, D.H. StJohn, M. Qian; The Effect of Manganese on the Grain Size of Commercial AZ31 Alloy; Materials Science Forum, 2005, Vol. 488-489, pp. 139-142
4] T. Laser, Einfluss von intermetallischen Phasen in der Magnesiumknetlegierung AZ31 auf Rekristallisation, Texturausbildung und mechanische Eigenschaften, Physical thesis, Hamburg, 2008
5] Y. Wang, S. Bong Kang, J. Cho, Microstructure and mechanical properties of Mg-Al-Mn-Ca alloy sheet produced by twin roll casting and sequential warm rolling, Journal of Alloys and Compounds 509 (2011), pp. 704-711
6] M. Ullmann, M. Oswald, S. Gorelova, R. Kawalla,

H.-P. Vogt, Strip rolling technology of magnesium alloys, Steel research journal, Metal Forming (2012) pp. 855-858
7] W. A. Johnson, R. F. Mehl, Trans. Metall. Soc. AIME 135 (1939), pp. 416
8] R. Kawalla, G. Lehmann, T. Spittel, Umformtechnische Möglichkeiten der Werkstoffgestaltung im Zuge der Erzeugung und der Weiterverarbeitung von Flachprodukten, Tagungsband MEFORM 2000, TU Bergakademie Freiberg
9] T. Sakai, Hot Working and Recrystallization of Steels and Other Metals, in: Proc. of 7th Int. Symp. on Physical Simulation of Casting, Hot Rolling and Welding, ISPS (1997), pp. 141
10] T. Heller, H.-P. Hougardy, R. Kawalla, Rechnersimulation der Warmumformung und der Umwandlung am Beispiel der Warmbanderzeugung, Stahl und Eisen 116 (1996), Nr. 4, pp. 115
11] M. Sellars, J. Whiteman, Recrystallization and grain growth in hot rolling, Met. Sci., Vol. 13, 1979, pp. 187
12] P. D. Hodgons, R. K. Gibbs, A mathematical model to predict the mechanical properties of hot rolled C-Mn and microalloy steels; ISIJ International; Vol. 32; 1992, pp.1329

Comportamento a deformazione e sviluppo di microstruttura di nastri in lega di magnesio AZ31 prodotti mediante processo twin roll casting

Parole chiave: Magnesio e leghe - Deformazioni plastiche - Processi

Il presente lavoro descrivere il nuovo metodo energeticamente vantaggioso per la produzione su scala industriale di nastri di magnesio con spessore fino a 1,0 mm che si basa sul processo Twin Roll Casting (TRC - un processo di colata continua diretta all'interno di due cilindri controrotanti e raffreddati ad acqua) e sulla successiva laminazione, sviluppato presso l'Institut of Metal Forming della TU Bergakademie Freiberg, in collaborazione con il MgF Magnesio Flachprodukte GmbH di Friburgo (Germania).
È stata analizzata la programmazione del processo di laminazione, sulla bese del controllo del comportamento riguardo alla ricristallizzazione, per raggiungere un grano fine nel processo di laminazione per nastri in lega di magnesio AZ31. Il comportamento nella ricristallizzazione dinamica di questa lega durante la deformazione a caldo è stato determinato mediante l'ausilio di prove di deformazione da compressione planare e esame microscopico. Sono state analizzate l'influenza della temperatura, della deformazione e della velocità di deformazione sull'attivazione del processo dinamico di ricristallizzazione durante la deformazione. Il comportamento alla deformazione è stato anche simulato mediante modelli semi-empirici, che includevano coefficienti specifici relativi ai parametri di lavorazione (deformazione, velocità di deformazione e temperatura).