HOT TEARING SUSCEPTIBILITY OF Mg-5Nd-xZn ALLOYS

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Abstract

Magnesium-neodynium-zinc (Mg-Nd-Zn) alloys are promising candidates as creep resistant alloys. Further, Nd is a rare earth (RE) addition with lower solid solubility and a relatively lower cost. Hence, the use of such alloys may result in a feasible and cost effective alternative for enhancing Mg alloy use in high temperature applications. Nevertheless, studies on the castability of Mg-Nd-Zn alloys are lacking. As such, the aim of this research was to investigate the hot tearing susceptibility of Mg-5Nd-xZn (x = 0, 3, 5, 7 wt%) alloys during permanent mold casting. Specifically, a constrained-rod casting mold equipped with a load cell was used to characterize hot tearing severity and determine the onset temperature of hot tearing. The onset solid fraction of hot tearing was subsequently determined via thermodynamic software. The results suggest that hot tearing severity increased initially with addition of Zn (up to 5 wt%), but then decreased with further addition to 7 wt%. This was likely attributed to both the low onset solid fraction of hot tearing (i.e. 0.5) recorded for this alloy, which enabled enhanced feeding and opportunity to heal developing hot tears, as well as the divorced eutectic structure observed which may have facilitated late stage feeding of eutectic liquid and hence limit the alloy's susceptibility to hot

Introduction

Magnesium alloys are widely used in transport and consumer electronic applications to reduce the weight of components. In the past, automotive use of Mg was generally limited to a few select applications such as instrument panels, steering wheels and valve covers [1]. Nowadays, with the continuous development of Mg-RE alloy systems resulting in improved creep resistance and high strength at elevated temperatures, automotive use of Mg is increasing towards powertrain applications and further advancements are desired for future automobiles. However, one limiting factor remains the high cost of such RE elements.

Addition of Zn to Mg-RE alloys has recently been shown to reduce the amount of RE required for optimum alloy strength and ductility during hot rolling [2]. Such reduction in RE content significantly lowers the cost of these alloys, thereby making them more attractive for industrial applications. Moreover, Nd is a RE addition with lower solid solubility and a relatively lower cost [3]. Hence, the use of Mg-Nd-Zn alloys may result in a feasible and cost effective alternative for enhancing Mg alloy use in high temperature applications. Preliminary studies [4-6] have attempted to characterize the phase formation, microstructure development and mechanical properties of Mg-Nd-Zn alloys. Such alloys have been found to possess optimum hardness and strength after heat treatment due to the formation of plate-shaped GP zones and precipitates on prismatic planes of the Mg matrix [6]. Nevertheless, studies on the castability of such alloys are lacking. Castability of a new alloy must be at an acceptable level, as this is the major processing route for production of Mg alloy components [1]. As such, this research aims to characterize the hot tearing behavior of this alloy system.

Hot tearing is a common and severe defect occurring in alloys at late stages of solidification. Reviews carried out by Sigworth [7], Eskin [8] and Li and Apelian [9] detail the many theories and mechanisms attributed to the formation of such defects. It has been suggested that hot tearing is a complex phenomenon with many factors influencing their formation. An improvement in the understanding of such factors will help to develop methods towards preventing the formation of such defects in Mg alloys and thereby enhancing the use of Mg in industrial applications.

This research aims to characterize the hot tearing behavior of four Mg-5Nd-xZn (x=0,3,5,7 wt%) alloys during permanent mold casting. The load cell method was used to determine the onset temperature of hot tearing. Such a technique has shown success in investigating the hot tearing behavior of many Mg alloy systems [10-13]. Pandat thermodynamic software was used to estimate the onset solid fraction of hot tearing. Further, thermal analysis was carried out to understand the role of alloy solidification on hot tearing. Finally, scanning electron microscopy was used to characterize the microstructure of the alloys.

Experimental Procedure

Melting and Casting

Casting experiments were carried out at the Magnesium Innovation Centre (MagIC) at Helmholtz-Zentrum Geesthacht in Germany. The Mg-5Nd-xZn (x = 0, 3, 5, 7 wt%) alloys were prepared from pure (99.95%) Mg ingots with additions of pure (99.95%) Nd and pure (99.9999%) Zn. The alloys were cast into billets, which were then used for hot tearing experiments. All castings were made with virgin ingot material (i.e. no recycled alloy was used).

The alloys (in billet form) were melted under a protective atmosphere consisting of Ar + SF $_6$ gas in an electric resistance furnace. Approximately 350 g of the alloys were melted in a stainless steel crucible for each casting trial. Each alloy was poured with a superheat of 100 °C into a mold preheated to 250 °C. The resulting pouring temperatures were 740 °C, 730 °C, 730 °C and 725 °C for the Mg-5Nd, 3Zn-, 5Zn- and 7Zn-contaning alloys, respectively. Two repeat casting trials were carried out for each alloy.

Hot Tearing Apparatus

The hot tearing apparatus consisted of a constrained-rod casting (CRC) steel mold and a contraction force measurement system with a load cell, as shown in Figure 1. Details of the apparatus are provided elsewhere [14]. The load cell was connected to a data acquisition unit. The mold was comprised of two parts: a vertical sprue and a 148 mm long horizontal circular rod. The circular rod

was slightly tapered (from 12.5 mm to 10 mm diameter) to minimize the effect of friction from the mold way to the solidifying melt [12,13]. The principal function of the apparatus was to monitor the evolution of hot tearing in the casting using the measurement of contraction force in conjunction with the temperature during solidification.

Mold temperature was monitored via three K-type thermocouples inserted along three locations of the mold: one at the sprue-rod junction (T1) and two (T2, T3) along the horizontal rod (Figure 1b). The thermocouple at the sprue-rod junction was inserted into the casting cavity (to monitor temperature of the melt during solidification) while the remaining two thermocouples were not placed in contact with the casting rod, but strictly used to monitor the mold temperature. The method for which the mold was heated is described elsewhere [14]. The thermocouples were connected to a data acquisition unit. Upon pouring of the alloys the temperature and force data were collected and used to generate force-time and temperature-time plots, which enabled the determination of the onset of hot tearing.

The mold was coated with boron nitride to prevent corrosion of the steel mold into the Mg melt. The coating was removed and reapplied after each casting experiment. Two repeats were carried out for each casting trial to ensure repeatability of results. The reproducibility of the present apparatus is well documented and reported elsewhere [15].

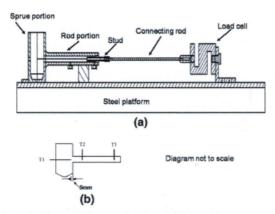


Figure 1. Schematic diagram showing a) CRC mold apparatus and b) location of thermocouples.

Microscopy

Representative samples were sectioned from the castings and subsequently mounted and ground using 500, 800, 1200 and 2500 grit SiC papers. The samples were then polished with 1 μm diamond suspension. The polished samples were viewed under a Tescan scanning electron microscope (SEM) to characterize the morphology and distribution of second phases.

Results and Discussion

Observation of Casting Surfaces

Upon completion of solidification, the castings were removed from the mold and examined for hot tears. The casting surfaces of

the investigated alloys are illustrated in Figure 2. A fine tear was present on the casting surface of the Mg-Nd alloy. Addition of 3 wt% Zn resulted in a significant increase in hot tearing severity, as shown in Figure 2b. In the case of the Mg-5Nd-5Zn alloy (Figure 2c), the hot tear was so severe that it resulted in the complete separation of the horizontal rod from the sprue. Finally, further addition of Zn to 7 wt%, was seen to significantly reduce the susceptibility of the alloy to hot tearing, as shown in Figure 2d

Onset of Hot Tearing

The force-time and temperature-time curves are presented in Figure 3 for the investigated Mg alloys. The thermocouple at the sprue-rod junction, T1, was used for temperature measurements. At the beginning of each trial, increased temperature in the temperature-time curve was coupled with a simultaneous increase in compressive load in the force-time curve for each condition. This was likely attributed to the filling of the casting cavity by molten metal. Initially, as the molten alloy entered the casting cavity and flowed toward the load cell, the pressure against the load cell generated a compressive load. Upon filling the casting cavity, solidification commenced and the casting began to contract with partial restriction (from the steel stud attached to the load cell), thereby changing the path of the force curve towards the opposite direction (tensile magnitude). The load continued towards a tensile state until a relief occurred. This relief in contraction force was attributed to the formation of a hot tear. Following this relief, the load again increased towards tension as the casting continued to contract. In some cases, tensile load magnitude was not obtained during solidification, as a result of the initial high magnitude of compression induced by the incoming melt on the load cell.

The hot tears present on the casting surfaces (Figure 2) were confirmed by the dip in the force-time curves, as shown in Figure 3. In the case of the Mg-5Nd alloy (Figure 3a), two hot tearing initiation points were observed. This suggested that two separate hot tears occurred during solidification. The corresponding initiation temperatures for the first and second tear were 602 °C and 559 °C, respectively. The onset solid fractions of hot tearing were calculated via Pandat thermal database software. The plots of solid fraction vs. temperature are given in Figure 4 for each investigated alloy. In the case of the Mg-5Nd alloy, the corresponding onset solid fractions of hot tearing were 0.77 and 0.86 for the first and second tear, respectively. Hot tears are known to typically occur within the solid fraction range of 0.85-0.95 [8]. However, solid fractions as low as 0.78 have been reported [10], thus the results are consistent with literature.

The large tear present on the casting surface of the Mg-5Nd-3Zn alloy (Figure 2b) was in agreement with the force-time curve (Figure 3b), which depicted a significant drop in force magnitude. Since hot tearing is a stress-relieving process, the size of the dip in the force curve can be directly related to the propagation of the hot tear. The hot tear present on the surface of this alloy was significantly larger than that of the Mg-5Nd alloy, and thus and greater drop in force magnitude was observed. The hot tear initiation temperature was recorded at 592 °C. This corresponded to a fraction solid of 0.7.

In the case of the Mg-5Nd-5Zn alloy, the severe hot tear that resulted in the complete separation of the horizontal rod from the

sprue was consistent with the force curve (Figure 2c). An initial hot tear was seen to initiate at 576 °C and propagated for some time, as no significant increase in load was observed. From there, the compressive force magnitude was seen to decrease towards tension until a second tear occurred at 492 °C. This tear was severe enough to result in the complete separation of the horizontal rod from the sprue, as from that instant there was further development of force magnitude (i.e. the force curve remained stagnant). Such a trend is typical of the bar contracting freely (i.e. without restriction from the sprue). The corresponding solid fractions of hot tearing were 0.69 and 0.9 for the two tears, respectively.

Finally, the fine tear seen on the casting surface of the Mg-5Nd-7Zn alloy resulted in a minor drop in the force-time curve, as shown in Figure 3d. In comparison to the 3Zn- and 5Zn-containing alloys, the magnitude of tear propagation (i.e. force drop or stagnant force development) was significantly lower. The hot tear was found to occur at 592 °C at an estimated solid fraction of 0.5. Thus, the tear was found to occur quite early during solidification. In turn, this likely enabled more time for the initiated hot tear to heal by liquid metal feeding during the remainder of solidification, thereby resulting in a less severe tear for this alloy in comparison to the 3Zn- and 5Zn-containing alloys.

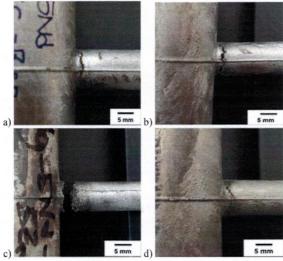
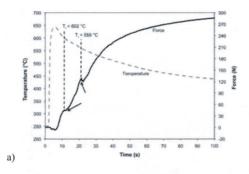
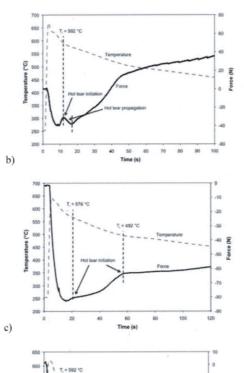


Figure 2. Presence of hot tears on surface of a) Mg-5Nd, b) Mg-5Nd-3Zn, c) Mg-5Nd-5Zn and d) Mg-5Nd-7Zn alloy castings.





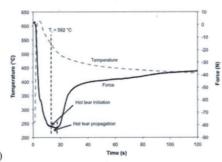


Figure 3. Force-temperature-time curves for the a) Mg-5Nd, b) Mg-5Nd-3Zn, c) Mg-5Nd-5Zn and d) Mg-5Nd-7Zn alloys.

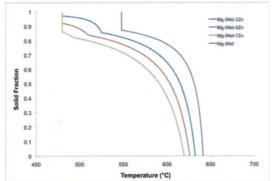


Figure 4. Solid fraction development for the Mg-5Nd-xZn alloys.

Thermal Analysis

Conventional thermal analysis was carried out from the temperature-time (cooling) curves of each alloy. The first

derivative of each cooling curve was taken and used to determine the freezing range, solidification time and the onset temperature of dendrite coherency. The onset temperature of dendrite coherency was determined from methods readily available in literature [16, 17]. The results of thermal analysis are summarized in Table I for the four Mg alloys. Further, differences in temperature (T) and time (t) between the points of interest are given in Table II. A discussion on the time and temperature intervals is provided and related to casting feedability.

Table I. Thermal analysis data for Mg-5Nd-xZn castings.

Alloy	Nucleation		Coherency		Solidification	
	T_N	t _N	T _C (°C)	t _C	T _S	t_{S}
5Nd	641	1.4	628	4.1	522	32.4
5Nd-3Zn	632	2.9	616	5.6	496	53.2
5Nd-5Zn	628	2.5	610	5.2	490	56.5
5Nd-7Zn	624	2.4	610	3.8	480	63.1

Table II. Temperature and time differences between points of

		mierest.		
Alloy	$t_C - t_N$	$T_N - T_S$	$(T_N - T_S)_S$	$t_S - t_N(s)$
	(s)	(°C)	(°C)	
5Nd	2.7	119	93	31.0
5Nd-3Zn	2.7	136	153	51.3
5Nd-5Zn	2.7	138	146	54.0
5Nd-7Zn	1.4	144	140	60.7

The duration of mass feeding of liquid metal was represented by the time difference between nucleation and coherency (i.e. $t_c - t_N$). The results suggest that minimal time was available for mass feeding in each alloy. This is consistent with the poor hot tearing resistance observed for all alloys (Figure 2). Further, the time for mass feeding was virtually identical for the Mg-5Nd, Mg-5Nd-3Zn and Mg-5Nd-5Zn alloys. In the case of the Mg-5Nd-7Zn alloy, however, the time interval for mass feeding was found to decrease to almost half with respect to the other alloys. Such an early onset of dendrite coherency in this alloy, may suggest the reason for which the hot tear occurred at such an early stage of solidification (0.5 solid fraction) with respect to the other alloys. Stress and strain begin to build within a casting after the dendrites become coherent and thus, an earlier development of stress and stain, likely may have lead to an earlier initiation of hot tearing in the Mg-5Nd-7Zn alloy.

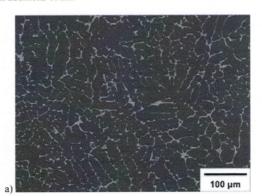
The temperature interval, $T_N - T_S$, represents the freezing range of the alloys, while the time interval $t_S - t_N$, represents the solidification time. The freezing calculated via Scheil using Pandat thermodynamic software is represented as, $(T_N - T_S)_{S.}$ A good agreement between the actual experimental values and those obtained thermodynamically was observed. Further, the results suggest that both freezing range and solidification time increased with the addition of $Z_{I.}$.

The increase in freezing range and solidification time was likely the reason for which the hot tearing severity was increased for the 3Zn- and 5Zn-containing alloys with respect to the Mg-5Nd binary alloy. It is well established that an increase in freezing range (and solidification time) often results in increased hot tearing susceptibility [8, 18]. However, in the case of the 7Zn-containing alloy, a further increase in freezing range and solidification time as well as the lowest time interval of mass

feeding was not consistent with the reduced hot tearing severity observed for this alloy (Figure 2). It is possible that the early initiation of hot tearing in this alloy enabled more time for (interdendritic) liquid metal feeding, as mentioned earlier. Another possible reason for this occurrence can be described by considering the work of Clyne and Davies [18]. The authors found that for many alloy systems, there reaches an amount of solute that results in a peak severity of hot tearing. Further increases in solute beyond the peak content, typically results in subsequent decreases in hot tearing severity. This trend is referred to as the lambda (λ) curve due to its shape. Thus, it may be the case that the addition of 7 wt% Zn to the binary Mg-5Nd alloy may be past the critical amount of Zn required for severe hot tearing. This may be due to an increased amount of eutectic liquid available for feeding (and subsequent healing of developing tears) at late stages of solidification as a result of the higher amount of solute. The microstructure of each alloy is presented in the following section.

Microscopy

Representative SEM micrographs are shown in Figure 5 for the investigated Mg alloys. Examination of the micrographs illustrates coarse dendrite morphology with distribution of second phases along interdendritic regions for each alloy. The second phase present in each alloy is likely either the Mg₃Nd phase. It has been shown that Mg-Nd casting alloys tend to contain Mg12Nd at relatively slow cooling rates or Mg3Nd if the cooling rate is particularly rapid, as in gravity die casting [19]. Further inspection of the micrographs suggests that the addition of Zn did not significantly increase the amount or affect the distribution of second phases in the microstructure. Thus, it cannot be suggested that a greater amount of eutectic liquid was present for healing the developing hot tear in the Mg-5Nd-7Zn alloy. The alloys were therefore examined at higher magnification in order to see whether changes in the morphology of the second phases occurred with additions of Zn.



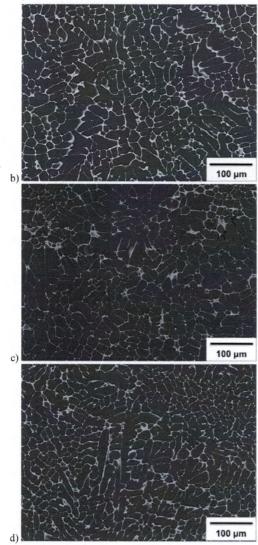


Figure 5. SEM micrographs of a) Mg-5Nd, b) Mg-5Nd-3Zn, c) Mg-5Nd-5Zn and d) Mg-5Nd-7Zn.

Three representative micrographs at higher magnification are illustrated in Figure 6 a, b and c for the Mg-5Nd, Mg-5Nd-5Zn and Mg-5Nd-7Zn alloys, respectively. In the case of the Mg-5Nd alloy, a fully divorced eutectic structure of the Mg₁₂Nd phase was observed as separate 'islands' of this phase was present along the Mg matrix. In contrast, a significant change in the morphology of the intermetallic was observed in the Mg-5Nd-5Zn alloy, as the structure appeared more plate-like. This plate-like structure was likely more brittle and thus more prone to hot tear formation. This was likely a significant factor affecting the alloy's resistance to hot tearing. Finally, in the case of the Mg-5Nd-7Zn alloy, shown in Figure 6c, the intermetallic appeared to become more divorced once again. The reason for this occurrence is not completely understood and subject to further investigation. Nevertheless, such larger 'islands' of intermetallic may suggest improved feeding

through larger channels at late stages of solidification, and thus an overall improved resistance to hot tearing.

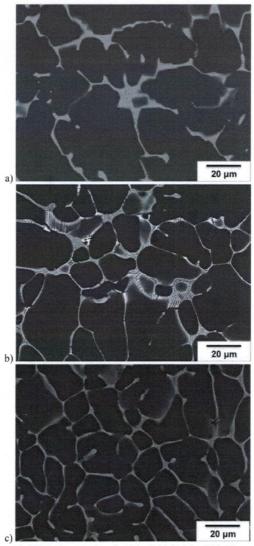


Figure 6. SEM micrographs of a) Mg-5Nd, b) Mg-5Nd-5Zn and c) Mg-5Nd-7Zn at higher magnification.

Conclusions

The hot tearing susceptibility of Mg-5Nd-xZn (x=0, 3, 5, 7) alloys was investigated during permanent mold casting. The following conclusions can be drawn from this study:

- Hot tearing was found to occur in all investigated alloys. Addition of Zn up to 5 wt% significantly increased the hot tearing severity. However, further addition of Zn to 7 wt% resulted in a decrease in hot tearing severity.
- The onset temperature of hot tearing was obtained from the force-time and temperature-time curves generated from the load cell. The hot tears for all alloys, with

- exception of the Mg-5Nd-7Zn alloy, were seen to occur at solid fractions ranging from 0.7-0.9. In the case of the Mg-5Nd-7Zn alloy, the onset solid fraction of hot tearing was recorded at 0.5.
- 3. Thermal analysis revealed an increase in freezing range and solidification time with addition of Zn. Such results are consistent with the increase in hot tearing severity observed for the Mg-5Nd-3Zn and Mg-5Nd-5Zn alloys with respect to the binary Mg-5Nd alloy. However, conflicting results were seen for the Mg-5Nd-7Zn alloy, as it was found to have the largest freezing range and longest solidification time but the lowest susceptibility to hot tearing of the three Zn-containing Mg-5Nd alloys.
- 4. Scanning electron microscopy illustrated a significant transformation in eutectic structure from fully divorced for the Mg-5Nd alloy to a more plate-like brittle structure for the Mg-5Nd-5Zn alloy. Such a brittle structure likely increased the alloy's susceptibility to hot tearing, thereby making it the most prone of the alloys investigated. In the case of the Mg-5Nd-7Zn alloy, however, the eutectic structure was again found to be more divorced, which in turn may have facilitated late stage feeding of eutectic liquid and thereby limit the alloy's susceptibility to hot tearing.

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