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**EVALUATION OF A CANADIAN PRODUCED
MAGNESIUM-NEODYMIUM MASTER
ALLOY FOR THE PREPARATION OF
QE22 CASTING ALLOY**

by

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PHYSICAL METALLURGY DIVISION

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EVALUATION OF A CANADIAN PRODUCED
MAGNESIUM-NEODYMIUM MASTER ALLOY
FOR THE PREPARATION OF QE22 CASTING ALLOY

by

B. Lagowski*

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SUMMARY OF RESULTS

Magnesium-Neodymium master alloy produced by Dominion Magnesium Limited was evaluated by comparison with a similar master alloy produced by Magnesium Elektron Limited, United Kingdom, and by comparison of the resultant properties of QE22 alloys produced using each of the master alloys. It was found that the DOMAL master alloy gave lower properties in QE22 alloys at room and elevated temperature, and this may have been due to the lower zirconium content of the alloy and/or to the different composition of the rare earths fraction of the alloy. Room temperature minimum properties required by specifications were easily obtained, but difficulties were encountered in meeting specification requirements for elevated temperature yield strength.

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INTRODUCTION

Magnesium casting alloy QE22 contains fractionated rare earths metals, composed predominantly of neodymium and praseodymium, and less desirable elements such as lanthanum and cerium. In addition to rare earths, this alloy contains silver and zirconium. At the present time, the rare earths metals are introduced in the form of Mg-RE master alloy, produced and distributed by Magnesium Elektron Limited (MEL), United Kingdom. An attempt was made by Dominion Magnesium Limited (DML), Haley, Ontario, to produce such master alloy for domestic use, and, in a letter dated February 10, 1966, ref: D/16/11/3 Nd. (Inquiry No. 1145), assistance was requested in the evaluation of this product.

It was proposed to make a series of small melts using master alloys produced by MEL and DML, the evaluation of the master alloys to be based on the following:

- (a) chemical analysis of the rare earths,
- (b) recovery of the rare earths in QE22 alloy,
- (c) room temperature properties of QE22 alloy, and
- (d) elevated (316°C) temperature properties of QE22 alloy.

MELTING PROCEDURE

A total of five melts of QE22 alloy were prepared, three using DML and two using MEL master alloys. Each 20-lb melt was prepared in steel pots using a gas-fired furnace. The following melting procedure was used: pure magnesium ingots were melted under 40-40-20 flux, and just before the last portion of magnesium was melted, the master alloy was charged on top of unmelted magnesium. When all the metal was melted, the temperature of the melt was raised to 760°C, silver was alloyed, the melt stirred for one minute, zirconium tetrachloride fused salt in the amount of 9½% of the charge was added, and the melt stirred for 10 minutes. After a settling period of 10 minutes, two green sand moulds of "Dow" type cast-to-shape test bars were poured at 760°C, yielding a total of 8 test bars.

RESULTS

(a) Chemical Analysis of the Master Alloys

Master alloy produced by DML was supplied with a spectrographic analysis of the various rare earths metals. Dominion Magnesium Limited also analysed, spectrographically, one of the QE22 alloys prepared using MEL master alloy. In addition, the Mines Branch Chemical Laboratory analysed, by the wet method, the iron content of both master alloys. The composition of the rare earths portion of the master alloys and the iron contents are presented in Table 1.

TABLE 1

<u>Master Alloy</u>	<u>La,%</u>	<u>Ce,%</u>	<u>Pr,%</u>	<u>Nd,%</u>	<u>Fe,%</u>	<u>Remarks</u>
DML	3.05	5.23	19.60	71.57	0.55	also contains Y
MEL	2.47	8.37	4.65	84.32	0.19	

(b) Recovery of the Rare Earths in QE22 Alloy

The recovery of the total rare earths in QE22 alloys prepared using both master alloys are given in Table 2.

TABLE 2

<u>Melt No</u>	<u>Master Alloy Used</u>	<u>RE in Charge %</u>	<u>RE in Alloy %</u>	<u>Recovery %</u>
5395	DML	2.93	2.12	72.4
5397	"	"	2.12	72.4
5399	"	"	2.17	74.0
5396	MEL	2.95	2.17	73.5
5398	"	"	2.20	74.5

The chemical analyses by wet methods of QE22 alloys as determined by the Mines Branch Chemical Laboratory are given in Table 3.

TABLE 3

<u>Melt No.</u>	<u>Master Alloy Used</u>	<u>RE, %</u>	<u>Ag, %</u>	<u>Zr sol., %</u>	<u>Zr insol., %</u>
5395	DML	2.12	2.57	0.49	0.07
5397	"	2.12	2.46	0.47	0.08
5399	"	2.17	2.64	0.48	0.08
5396	MEL	2.17	2.64	0.54	0.03
5398	"	2.20	2.46	0.55	0.04

(c) Room Temperature Properties

All test bars were heat treated as recommended by MEL, which consisted of solution heat treatment for 8 hr at 525°C, followed by quenching in water at 60°C, and ageing for 8 hr at 200°C. Three test bars from each melt were tested at room temperature and the average results are given in Table 4.

TABLE 4

<u>Melt No</u>	<u>Master Alloy Used</u>	<u>UTS</u> <u>kpsi</u>	<u>0.2% YS</u> <u>kpsi</u>	<u>Elong.</u> <u>%</u>	<u>Grain Size</u> <u>0.001 in.</u>
5395	DML	39.4	30.5	4.5	3.0
5397	"	38.7	29.0	3.5	3.0
5399	"	38.6	29.0	5.0	3.0
5396	MEL	41.4	31.6	5.0	3.0
5398	"	40.9	32.3	5.0	2.5
Minimum QQ-M-56b		35.0	25.0	2.0	

(d) Elevated Temperature Properties

Two test bars from each melt in the fully heat treated condition (T6) were tested at elevated temperature - 316°C (600°F) according to the requirements of QQ-M-56b specification, which states that: "Tensile test specimens shall be heated to 600°F (316°C) ±5°F, held for 10 minutes before testing and loaded at this temperature at a rate of 0.005 inch per inch per minute up to the yield strength and at a rate of 0.10 inch per inch per minute above the yield strength". During the heating period, it was found that the furnace overshoots at this testing temperature and it was necessary to use semiautomatic temperature control until the testing temperature was reached. This procedure, however, prolonged the heating time and made it inconsistent from test to test. It is believed that although the heating rate is not specified in QQ-M-56b it may have pronounced effect on the results. The strain rate of 0.005 inch per inch per minute was controlled by a strain pacer connected with an extensometer. The strain rate above the yield was controlled by a load rate pacer.

The results obtained on each separate test bar are presented in Table 5.

TABLE 5

Melt No	Master Alloy Used	Bar No	Properties at 316°C				Heating Time, Minutes	
			UTS kpsi	0.2% YS kpsi	El. %	RA %	200°C-316°C (390°F-600°F)	
5395	DML	12	12.45	9.22	59.0	86.2	*	
		42	14.45	10.30	52.5	83.8	40	
5397	"	21	13.50	9.60	60.0	83.0	45	
		22	13.52	9.40	54.0	81.4	60	
5399	"	11	13.32	9.36	55.0	77.8	50	
		31	13.91	10.04	67.0	83.0	30	
5396	MEL	41	15.30	11.24	49.5	83.3	30	
		32	14.12	10.20	51.0	83.0	45	
5398	"	11	13.65	9.70	55.0	77.8	70	
		31	13.11	10.48	67.0	83.0	30	
Minimum QQ-M-56b			13.00	10.00				

* Uncontrolled rate of strain

DISCUSSION OF RESULTS

Basic differences in the chemistry of the rare earths metals of the two master alloys are shown in the lower content of Ce and Nd but higher content of Pr in the DML master alloy. Also the DML master alloy contains yttrium metal which was not detected in MEL master alloy. The relative content of rare earth elements in the master alloy is essentially carried over to the QE22 alloys produced from respective master alloys. However, slight preferential loss of certain rare earths elements occurs during preparation of QE22 alloy due to reaction between $MgCl_2$ formed by reduction of $ZrCl_4$ with magnesium and rare earths elements. For example, the total rare earths in the QE22 alloy was decreased from 2.14% to 1.35% by triple application of $ZrCl_4$ fused salt, but at the same time, the Ce and La increased while Pr and Nd decreased in the rare earths fraction of the alloy. This means that a slight enrichment in the relative La and Ce content of the rare earths metals may be expected in the alloy prepared from recirculated scrap. The higher iron content in the DML master alloy is to be noted and it is believed that this is responsible for the lower soluble and higher insoluble zirconium content in the respective QE22 alloy. The differences in the chemistry of master alloys did not, however, affect the recovery

of total rare earths in QE22 alloy. The room temperature properties of QE22 alloy, prepared using DML master alloy, are lower than those of the alloy prepared from MEL master alloy but in both cases the specification minima are met easily. It is believed that the lower "soluble" zirconium content of the alloy prepared from the DML master alloy is responsible for the decrease in properties.

Elevated temperature properties of QE22 alloy, prepared from DML master alloy, were generally lower than those obtained on alloy produced from MEL master alloy. This is particularly evident in results for yield strength. With the exception of one test bar, all results for the ultimate tensile strength were higher than the specification minimum, but in the case of the yield strength only two results out of six in the DML alloy and three out of four in the MEL alloy were higher than the specification minimum. It is interesting to note that the highest yield strength was obtained on test bars heated to testing temperature for the shortest time. This is due to the rapid overageing of the alloy at high temperature of approximately 300°C (575°F).

In order to collect more information on the effect of heating time on elevated temperature properties Mr. A.A. Moore of Dow Company was asked for advice in this matter. His reply indicates that the heating time may have a pronounced effect on properties but also that the specification minima are set too high and that a revision of AMS 4418 is now out for vote. In the new version, the properties are lowered to 11,500 psi tensile and 9,000 psi yield strength. The loading rates are changed to be not greater than 0.05 inch per inch per minute up to yield and between 0.11 and 0.14 inch per inch per minute above the yield strength. If the specification requirements would be adopted, all test bars would pass the specification minima. The reason for the lower elevated temperature properties of the alloy prepared from DML master alloy may also be due to the lower zirconium content and/or due to different composition of the rare earths portion of the alloy.

CONCLUSIONS

1. Magnesium-neodymium master alloy produced by DML, when compared with that produced by MEL, contains higher iron content and shows the presence of yttrium, which was not detected in MEL master alloy. The composition of rare earths in the DML master alloy also shows higher Pr, but lower Nd and Ce contents.

2. Recovery of rare earths in the QE22 alloy is constant regardless of the master alloy used.
3. Melts of QE22 prepared from DML master alloy show a lower soluble zirconium content which is thought to be mainly responsible for slightly lower room temperature properties although these materials still comfortably meet the specification minima.
4. Elevated temperature properties of QE22 prepared from DML master alloy are lower and difficulties may be encountered in meeting specification requirements for the yield strength. The lower zirconium content and/or different composition of the rare earths fraction may be responsible for these lower properties. However, if the proposed revised specification were adopted, no difficulties would be expected in meeting its requirements.

BL/sg