



Development and Flammability Testing of Magnesium Alloys for Space Applications

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What and Why?

WHAT?

- This project tested and compared the flammability of select Mg alloys for use in a simulated ISS environment.
- Factors that influence flammability of selected alloys in 24.1% oxygen were analyzed.

WHY?

- Mg is lightweight while retaining strength
- To address negative perceptions regarding the flammable nature of Mg.
 - A restriction on using Mg alloys has been due to a negative view of Mg in terms of flammability and corrosion.
- Because investigating flammability of materials is crucial at NASA.
 - Investigating flammability is important at NASA, as seen from the Apollo 1 crew cabin fire.
- To keep innovation on par with commercial companies and other government organizations.
 - Commercially, Mg alloys are used on aircraft engines such as the Rolls-Royce RB211, RB. 183 Tay, and the BR710.
 - The military uses Mg alloys in engine gearboxes on helicopters.
 - Within the last five years, the FAA lifted a ban on the use of certain Mg alloys after conducting full-scale flammability simulated tests (video).
- To conduct the first flammability testing in oxygen enriched environments on selected magnesium alloys.
 - Occupational Safety and Health Administration (OSHA) has specified enriched oxygen concentrations to be above 22%.
- In keeping true to NASA's mission pioneering the future in scientific discovery and research.



FAA testing on WE43 in air. Video provided by Luxfer MEL Technologies.



What are the Current Restrictions on Using Mg at NASA?

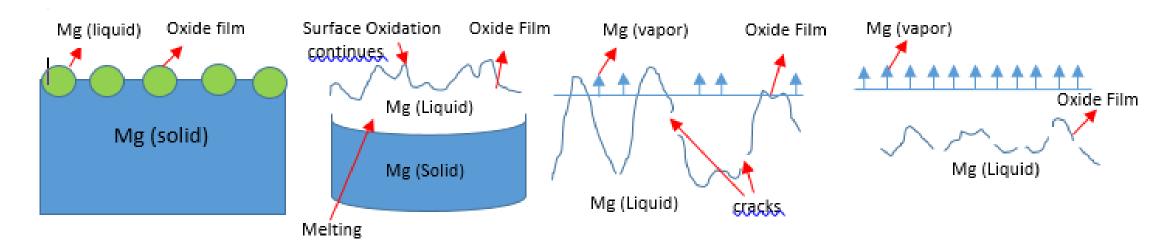
In 2016 the NASA-STD-6016A Standard Materials and Processes Requirements for Spacecraft was updated to remove a previous restriction on Mg alloy use.

4.2.2.4 Magnesium

- a. Magnesium alloys shall not be used except in areas where minimal exposure to corrosive environments can be expected and protection systems can be maintained with ease and high reliability.
- b. Magnesium alloys shall not be used in primary structure or in other areas of flight hardware that provides mission-critical functions that are subject to wear, abuse, foreign object damage, abrasion, erosion, or at any location where fluid or moisture entrapment is possible.
- c. Magnesium alloys shall not be machined inside spacecraft modules during ground processing or in flight, because machining operations can ignite magnesium turnings and cause fire.



Mg Ignition Mechanism



Temperature Increase: Surface oxidation reaction causes an increase in localized heat Temperature reaches boiling point of Mg. Vapor pressure build up causes an eruption of liquid through cracks of MgO.

Ignition is cause by the reaction of vapor with oxygen atmosphere.



Material Selection

Selection of Mg alloys:

- Selected Mg alloys WE43 and EV31 both contain rare earth(RE) elements.
- When testing in air, RE elements are know to form a stable oxide on the surface of the material restricting further oxidation.

• Selection of comparison material:

- Magnesium comparison: AZ31
 - Both Zn and Al are reported to lower ignition temperature.

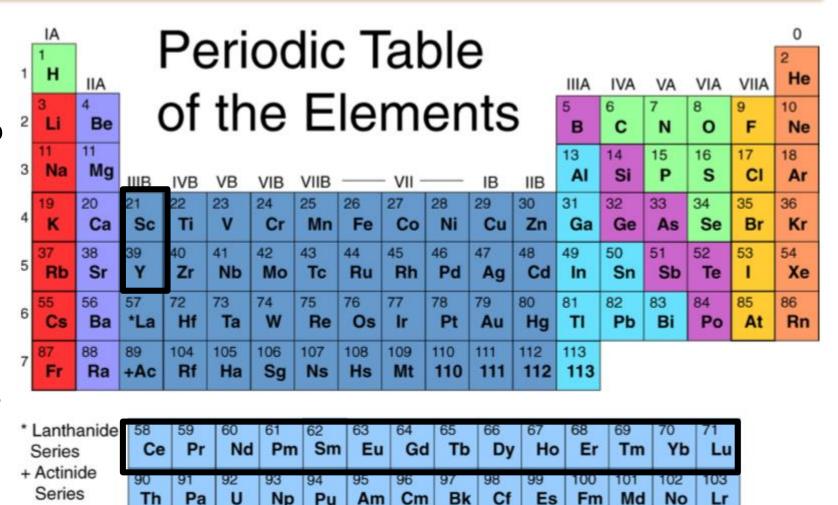
• Obtained Material:

 All material was procured by Luxfer MEL Technologies, along with a Certificate of Conformance (COC).



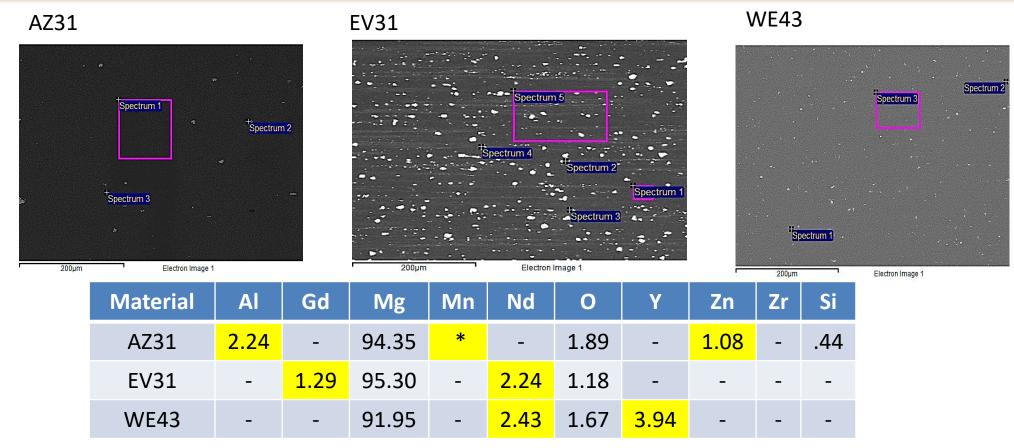
Back to Basics

- Elements are added to Mg in order to enhance strength and resistance to corrosion and flammability.
- Selected alloys are made adding up to 4% of RE elements to Mg.
- Yttrium (Y), Neodymium (Nd), Gadolinium (Gd) are highly reactive and have low solubility in Mg.





Compositional Analysis of Selected Alloys using SEM/EDS



^{*}In AZ31, Mn was detected as particulates in spectrum 2 and 3, not spectrum 1.

• Semi-quantitative compositional results given from a representative spectrum taken from each alloy using EDS at KSC. Values are taken from spectrum 1 on AZ31, spectrum 5 on EV31, and spectrum 3 on WE43. Highlighted values include those which influence ignition mechanism.



Flammability Testing

•NASA-STD-6001A, Test 17 "Upward Flammability of Materials in Gaseous Oxygen (GOX)" was conducted at WSTF.

-This test refers to ASTM G124 "Standard Test Method for Determining the Combustion Behavior of Metallic Materials in Oxygen-Enriched Atmospheres."

• Test was modified to be conducted at 24.1% oxygen and 14.7 psi to simulate the environment inside the

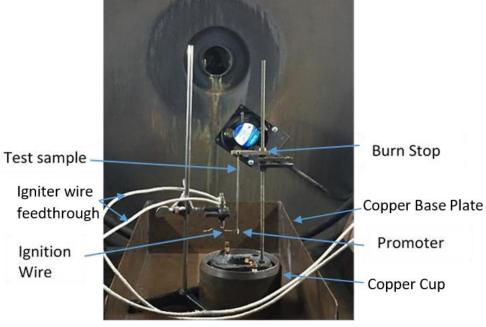
Ignition

Wire

International Space Station crew cabin.



Test chamber at WSTF



1/4" 3/16" 1/8"

Test sample set-up. One rod of each of the three different Mg alloys were tested at 4 different rod thicknesses.



Test Modifications

NASA STD 6001A, Test 17 Standard Test	Test modifications	Rationale for modification
Testing at 100% oxygen and various pressures to reach threshold pressure	- Test at 24.1% oxygen and 14.7 psi	- In order to simulate conditions present in the ISS crew cabin
Testing sample has a 1/8" diameter	Test at different thicknesses including: - 1/8", 3/16", 1/4", 3/8"	To determine threshold for future applications based on thickness



Results: Overview

- All 1/8" and 3/16" Mg alloys burned fully.
- AZ31 1/4" burned fully, while WE43 and EV31 had a lower burn length.
- 3/8" rods did not ignite.





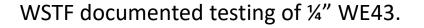
Results: Mg Alloy Comparison

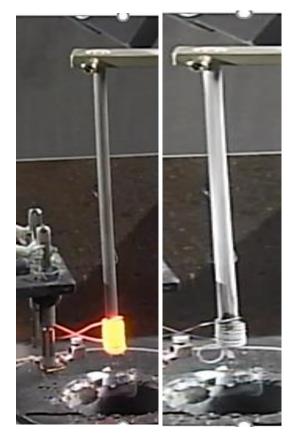
Material Name	Diameter	Burn Length	Time to Ignition from application of current	Temp @ ignition 1.2 in from coil	 This table provides a representative set of data from the tested Mg alloys. Valid ignition temperatures were
	(in.)	(in)	(m:s)	(°F)	only given for 0.25 inch
Mg alloy AZ31	0.25	5.1	1:10	622	rods. Thinner rods fully burned and thicker rods did not burn.
Mg alloy EV31	0.25	4.6	0:52	607	
Mg alloy WE43	0.25	3.6	1:28	649	Best performing alloy



Results: Formation of White Oxide Layer in WE43



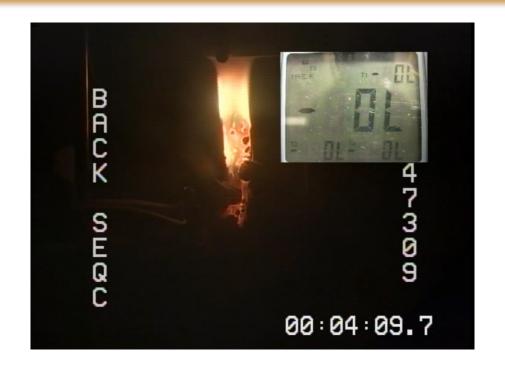


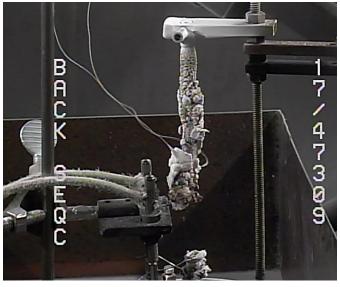


Observation: A white oxide was formed after burn.



Results: Formation of Nodules in AZ31





Observed nodules on post tested material.

WSTF documented testing of AZ31.

• The significance of these results is the fact that in AZ31, no insulating layer is formed and instead nodules are formed.



KSC Analysis Approach

- Conduct lab analysis at KSC to determine mechanism.
 - Non-destructive analysis:
 - Visual inspection and dimensional analysis
 - Computed tomography (CT) analysis
 - Optical microscopy
 - Scanning electron microscopy (SEM)
 - Chemical analysis using energy dispersive spectroscopy (EDS)
 - Destructive Analysis
 - Prepared samples using metallographic techniques to obtain cross section.
 - Metallography and microstructural evaluation of prepared samples.



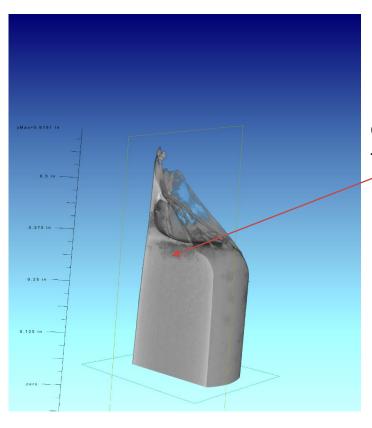
Computed Tomography (CT)

- CT was conducted to determine what area to analyze as a cross section with the SEM.
- Based on CT images, we observed a less dense region in a longitudinal section at the top of the rod.
- This area was selected for cross sectioning as a possible heat affected zone (HAZ).



WE43 rod analyzed with CT.

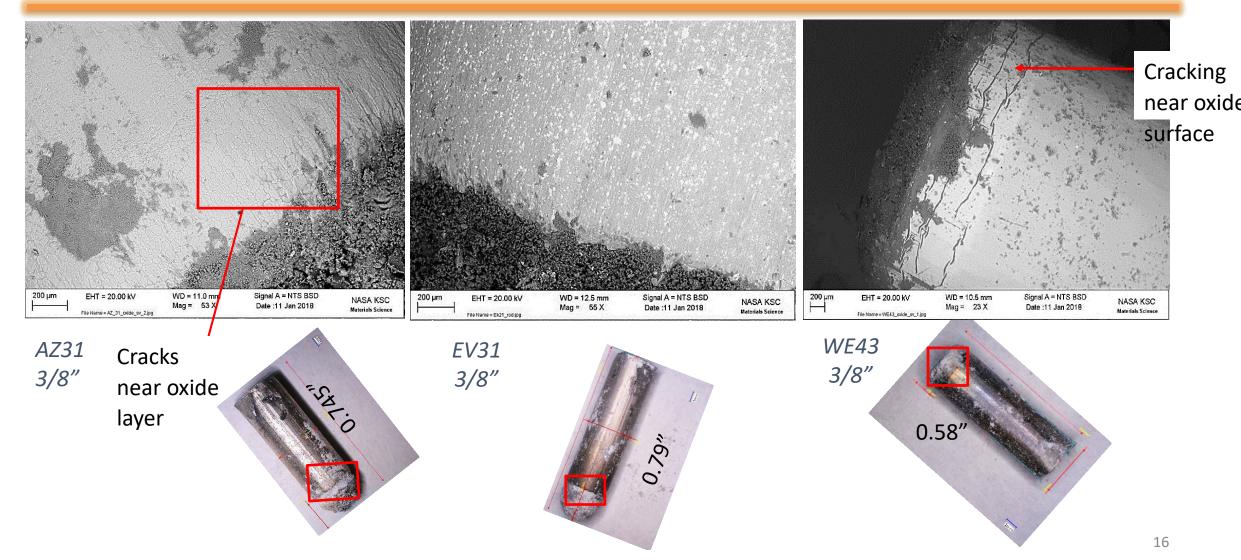
Red dotted lines indicates
areas of transverse sectioning
on CT images.



Less dense area corresponding to a HAZ



Surface Analysis of rods with SEM



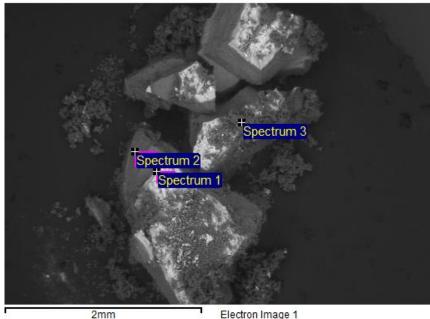


Analysis of Oxides with SEM/EDS/XRD

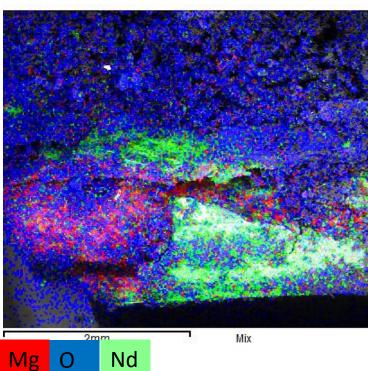
EV31

WE43









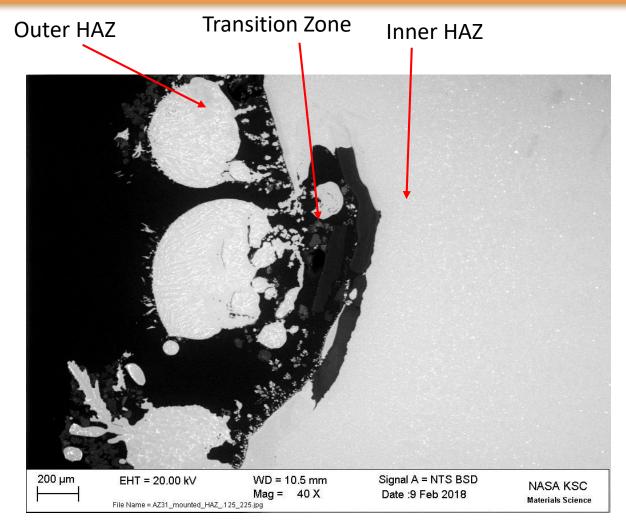
EDS spectrum 1 showed presence of Y, Nd, Dy, Mg and O. Approximately 30 wt.% Y, 1.9 wt.% Nd, and 1.3 wt.% Dy.

- Oxides are composed of rare earth elements that have low solubility in Mg at high temperatures.
- X-ray Diffraction confirmed presence of Nd_{1.6} Y_{.4} Zr_{.2} O_{.7} and MgO for WE43.



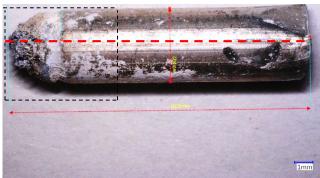
Cross Section for 1/8" Rod: AZ31







Cross Section for 3/16" Rod: AZ31



Sample was cross sectioned longitudinally and then cold mounted. Scale as shown.



Stitched image taken from tip of sample using digital microscopy.

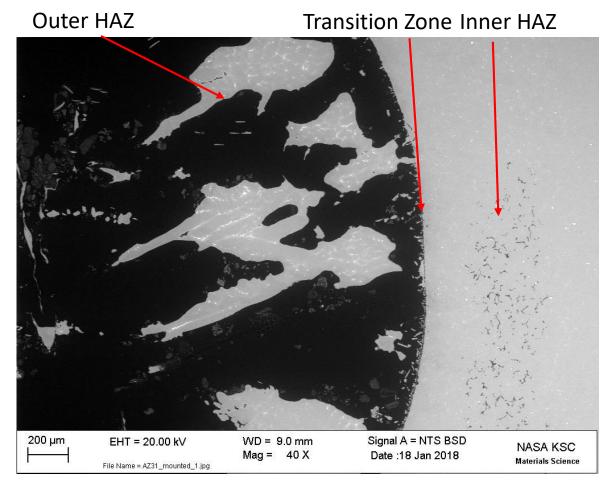
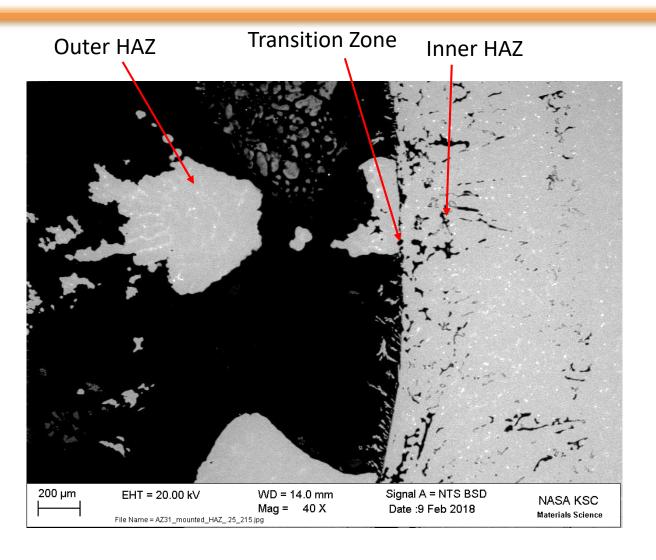


Image taken using the SEM



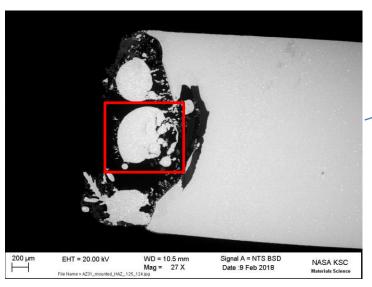
Cross Section for 1/4" Rod: AZ31



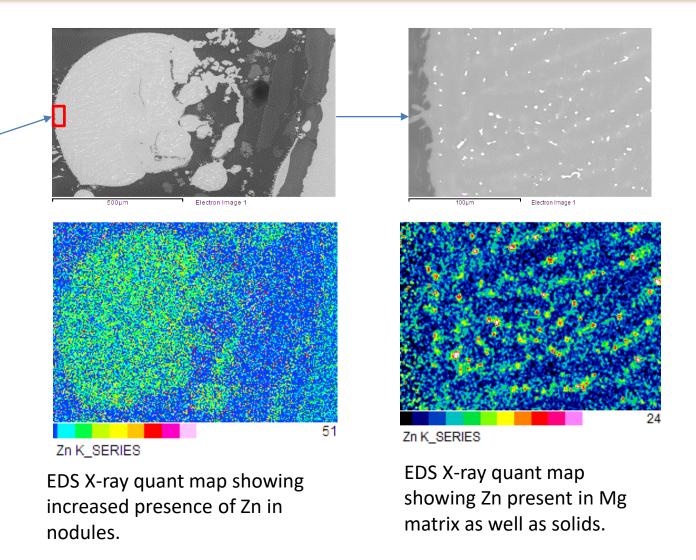




X-ray dot maps showing presence of Zn in AZ31 nodules



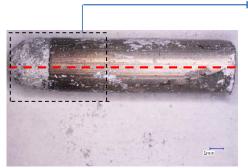
SEM image on 1/8" AZ31 HAZ showing formation of nodules.





Cross Section for 3/16" Rod: EV31

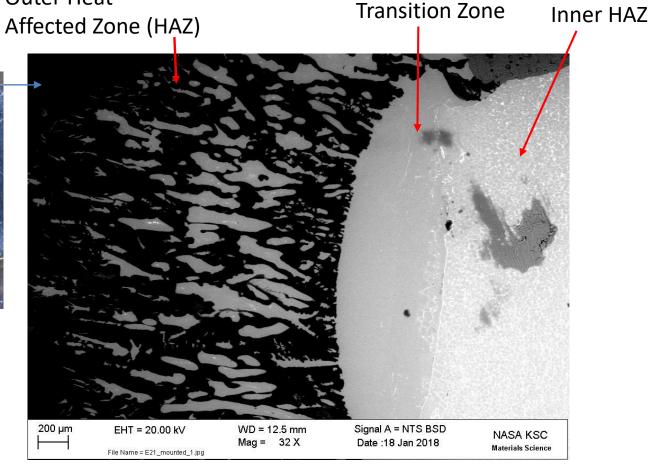
Outer Heat



Sample was cross sectioned longitudinally (red line) and then cold mounted. Scale as shown.

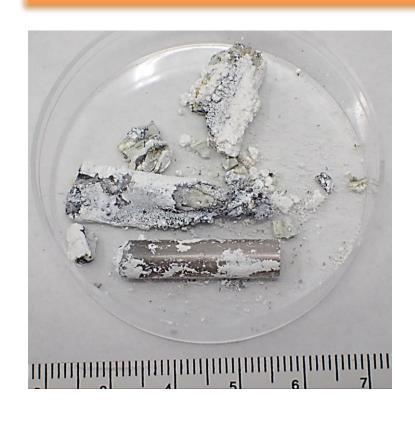


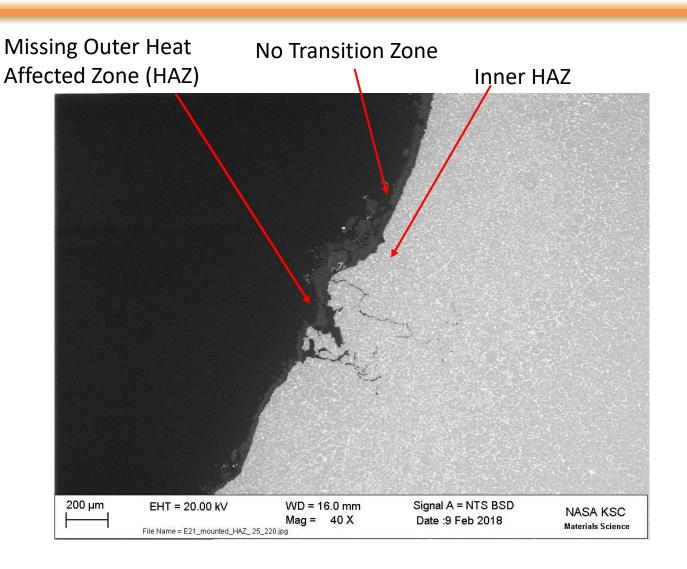
Stitched image taken from tip of sample using digital microscopy.





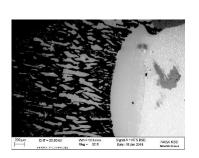
Cross Section for 1/4" Rod: EV31

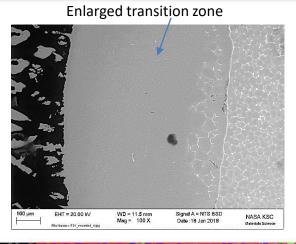


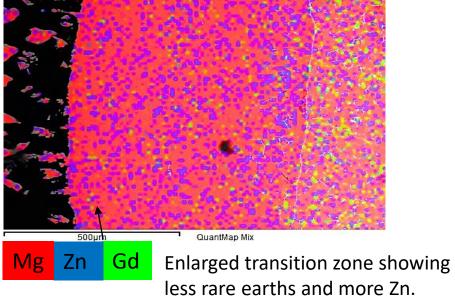




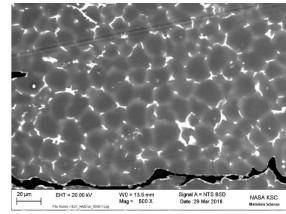
EV31 X-ray Quant Maps

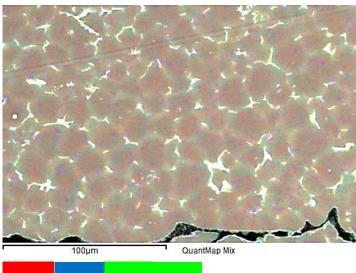










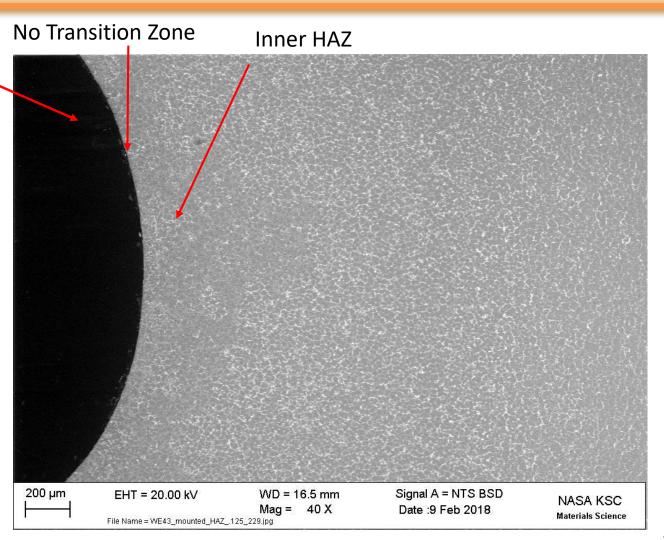




Cross Section for 1/8" Rod: WE43

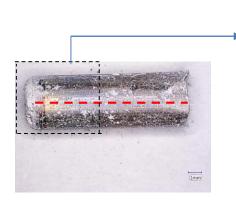
Missing Outer Heat Affected Zone (HAZ)







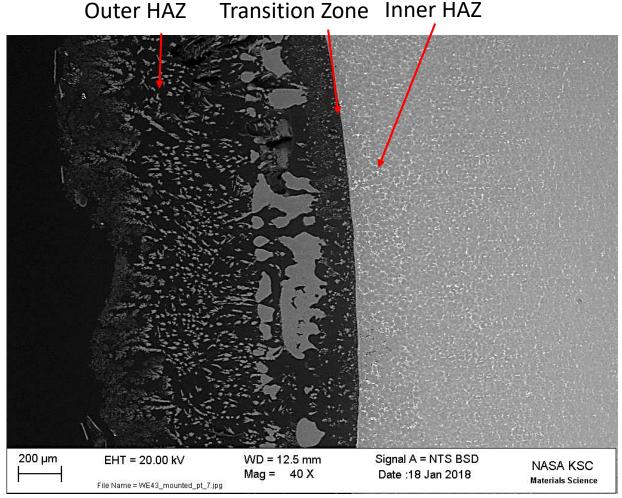
Cross Section for 3/16" Rod: WE43



Sample was cross sectioned longitudinally and then cold mounted. Scale as shown.



Stitched image taken from tip of sample using digital microscopy.



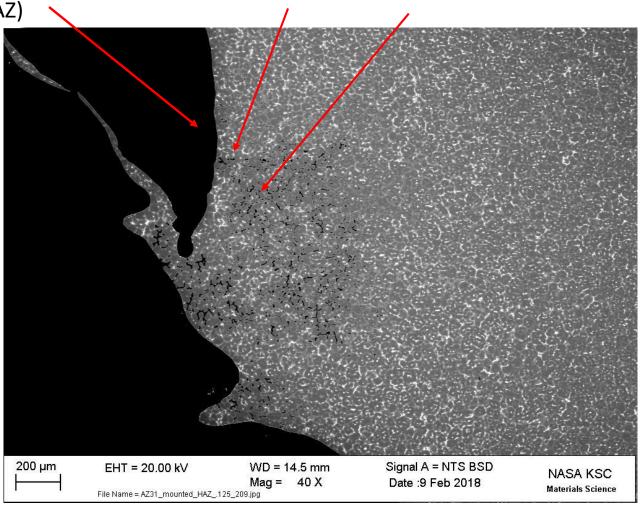


Cross Section for 1/4" Rod: WE43

No Transition Zone

Missing Outer Heat Affected Zone (HAZ)



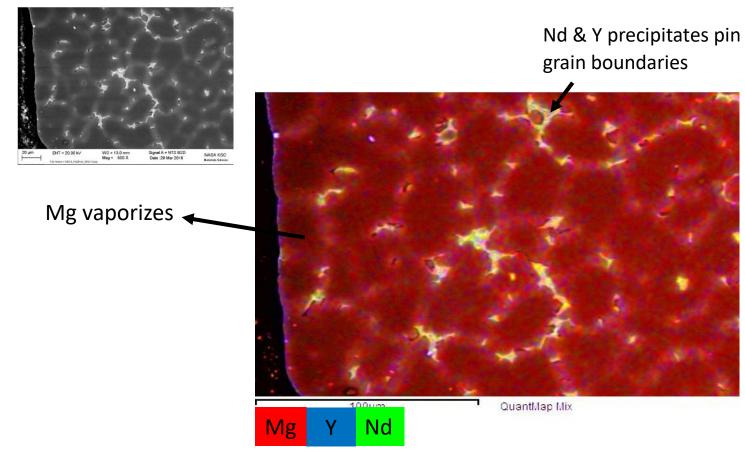


Inner HAZ



Proposed mechanism for oxide formation in WE43





This SEM image taken of the inner HAZ of WE43 shows the proposed mechanism of the formation of the oxide layer composed of RE elements. As the Mg vaporizes, the Y and Nd remain intact and combine with O₂ on surface to form an oxidation layer.



Etched Samples for 1/8" rod: Comparison of Grains in Inner HAZ

WE43

Particle Counts were

E112 "Standard Test

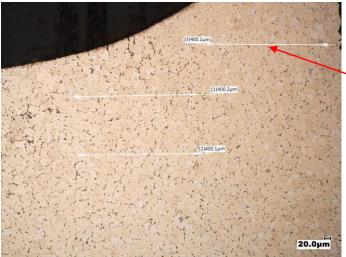
Average Grain Size"

intercept method.

taken per ASTM

Methods for Determining

using the line



[1]400.1µm

Average grain size = 80 um

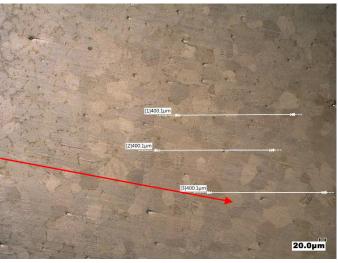
Average grain size = 22.2 um

WE43 appears to have smaller grains compared to AZ31, which validates proposed mechanism of Y & Nd pinning the grain boundaries.

Average grain size = 50 um

Average grain size = 19.0 um





AZ31



Summary of Observations

- Increasing rod thickness decreased burn length for all tested alloys.
 - At 3/8" thickness none of the three tested Mg alloys burned.
- Flammability tests results showed WE43 outperformed EV31 and AZ31.
- Videos showed white oxide layer was formed on EV31 and WE43, while nodules were formed on AZ31.
- For WE43:
 - EDS showed it contained Nd and Y segregated in grain boundaries.
 - XRD/EDS showed oxide layer was composed $Nd_{1.6} Y_4 Zr_2 O_7$ and MgO.
- For EV31:
 - Contained an enlarged transition zone.
 - This zone contained less RE elements and more Zn.
 - EDS showed oxide layer contained RE elements, as seen for WE43.
- For AZ31:
 - Nodules formed in HAZ contained Zn along grain boundaries with a Mg matrix.
 - Zn appeared to flow along columnar grain boundaries.
- For Etched samples:
 - Smaller grains in WE43 as compared to AZ31.







Conclusions from Observations

- Significance of study:
 - No previous flammability testing of Mg alloys have been conducted at 24.1% oxygen concentrations.
 - Adding to knowledge of lightweight Mg alloys for possible future applications.
- Thickness was crucial in increasing the flammability resistance.
- Adding Y & Nd was important in increasing the flammability resistance of WE43 tested at 24.1% oxygen.
- Y & Nd forms a uniform insulating oxide layer on surface of WE43.
- Adding Zn seems to decrease the flammability resistance in AZ31 & EV31.
- Nodules formed on the surface of AZ31 did not provide same resistance as insulating oxide layer.
 - Grain coarsening shows greater heat effects on AZ31.

39 Melting Temp: 2,779 F

Nd

144

Melting Temp:

1.861 F

60

Low solubility in Mg

High affinity to oxygen

30 Melting Temp: 787.5 F

High solubility in Mg





Forward Work

- Additional flammability testing:
 - At 24.1% oxygen to get statistically significant results.
 - At 30% and 35% oxygen to simulate deep space exploration crew environments.
 - Samples with anodic surface finishes.
- Collaboration with experts in the industry, academia, and government to continue work:
 - Publishing results in scientific journal.
 - Develop advanced Mg alloys with University of Florida.
- Targeting specific flight hardware applications for Mg alloys.
- Completing corrosion testing for internally NASA funded project.
- Using modeling techniques to substitute Mg for applications in spacecraft design.
- Conducting other types of materials tests for payloads exposed to low-earth orbit environments (atomic oxygen, radiation and thermal fluctuations).



Acknowledgements

- NASA for providing project funding and support.
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- NASA KSC contributors for providing technical expertise.



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Questions?



BACK-UP



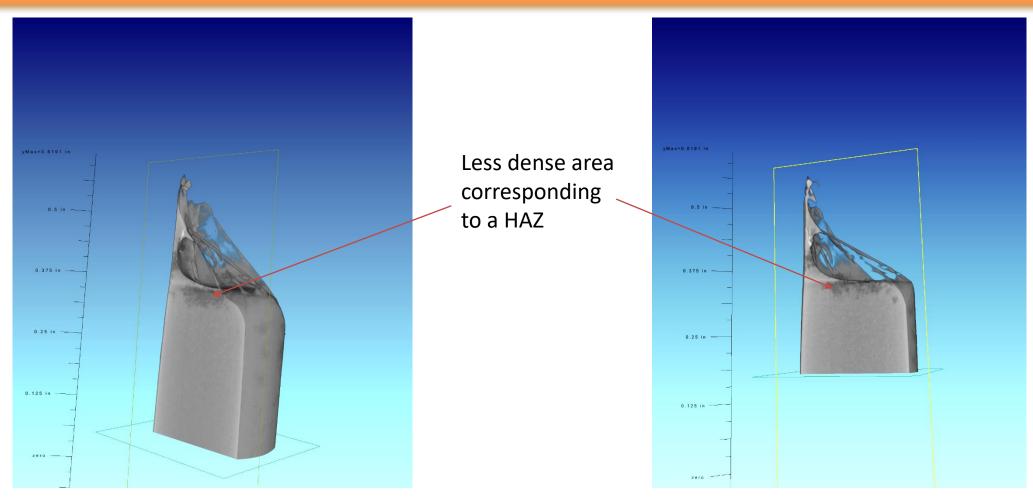
Organization Overview

- Laboratories, Development & Testing Division (NE-L):
 - Provides scientific and engineering services to NASA and contractor customers at KSC as well as to outside organizations.
 - Provides unique solutions to urgent problems in support of Commercial Crew Program (CCP), Exploration Ground Systems (EGS), Launch Services Program (LSP), International Space Station (ISS) and research and development projects.
 - Specialties include testing & design, fabrication & development, analytical laboratories, materials science, exploration payloads, advanced engineering development.
- Materials Science Branch (NE-L4):
 - Materials Testing and Failure Analysis
 - Corrosion Testing and Engineering
 - Materials & Processes (M&P) Engineering





Possible HAZ on CT



Additional images from CT conducted on WE43 emphasizing region of interest.



White Sands Data Sheet

WSTF No.	Material Name	Diameter	Burn Length	I	gniter Configuration		Time To Ignition from application of current	Temp @ ignition 3 cm from coil*
NO.		(in.)	(in)	Promoter	Igniter	Current Profile	(s)	(F)
		0.125	5	Mg ribbon hooked around bottom of sample	7 wraps 20 ga Kanthal wire	10 A until ignition + 15s	0:27	NR
	Magnesium Alloy WE43B (welding rod W27,	0.1875	5.1	Mg ribbon hooked around bottom of sample	7 wraps 20 ga Kanthal wire	10 A until ignition + 15s	0:36	NR
17-47307	SAE AMS4393, SAE AMS4427, BS ISO 3116:2007)	0.25	3.58	4 Mg ribbon Streamers	11 wraps 20 ga Kanthal wire	7A/40s, 10A/30s, 7A until ignition + 15s	1:28	649
		0.375	NI	End of rod countersunk and slots cut out of remaining wall plus 4mg ribbon stremmers	11 wraps 20 ga Kanthal wire	7A/40s, 10A/30s, 7A until ignition + 15s	N/A	NR
		0.125	4.88	Mg ribbon hooked around bottom of sample	7 wraps 20 ga Kanthal wire	10 A until ignition + 15s	0:33	NR
		0.1875	5	Mg ribbon hooked around bottom of sample	7 wraps 20 ga Kanthal wire	10 A until ignition + 15s	0:33	NR
17-47308	Magnesium Alloy Elektron 21 (EV31A, welding rod W28, SAE AMS4429A, SAE AMS4391)	0.25	4.61	4 Mg ribbon Streamers	11 wraps 20 ga Kanthal wire	7A/40s, 10A/30s, 7A until ignition + 15s	0:52	607
		0.375	NI	End of rod countersunk and slots cut out of remaining wall plus 4mg ribbon stremmers	11 wraps 20 ga Kanthal wire	7A/40s, 10A/30s, 7A until ignition + 15s	N/A	NR
		0.125	4.84	Mg ribbon hooked around bottom of sample	7 wraps 20 ga Kanthal wire	10 A until ignition + 15s	0:45	NR
	Magnesium Alloy AZ31 (ASTM B107)	0.1875	5	Mg ribbon hooked around bottom of sample	7 wraps 20 ga Kanthal wire	10 A until ignition + 15s	1:00	NR
17-47309		0.25	5.12	4 Mg ribbon Streamers	11 wraps 20 ga Kanthal wire	7A/40s, 10A/30s, 7A until ignition + 15s	1:10	622
		0.375	NI	End of rod countersunk and slots cut out of remaining wall plus 4mg ribbon stremmers	11 wraps 20 ga Kanthal wire	7A/40s, 10A/30s, 7A until ignition + 15s	N/A	NR



3/16" rod AZ31 etch



Dark grains are likely Mg₂Si particles that did not dissolve

Uneven grain boundaries are caused by a small amount of unresolved discontinuous precipitate formed even upon rapid cooling from solution temperature



ASTM E112 for Etched Samples



TABLE 4 Grain Size Relationships Computed for Uniform, Randomly Oriented, Equiaxed Grains

Grain Size No.	\bar{N}_A Grains/Unit Area		Ā Average (A Average Grain Area		d Average Diameter		₹ Mean Intercept	
G	No./in.2 at 100X	No./mm ² at 1X	mm ²	μm²	mm	μm	mm	μm	No./mm
00	0.25	3.88	0.2581	258064	0.5080	508.0	0.4525	452.5	2.21
0	0.50	7.75	0.1290	129032	0.3592	359.2	0.3200	320.0	3.12
0.5	0.71	10.96	0.0912	91239	0.3021	302.1	0.2691	269.1	3.72
1.0	1.00	15.50	0.0645	64516	0.2540	254.0	0.2263	226.3	4.42
1.5	1.41	21.92	0.0456	45620	0.2136	213.6	0.1903	190.3	5.26
2.0	2.00	31.00	0.0323	32258	0.1796	179.6	0.1600	160.0	6.25
2.5	2.83	43.84	0.0228	22810	0.1510	151.0	0.1345	134.5	7.43
3.0	4.00	62.00	0.0161	16129	0.1270	127.0	0.1131	113.1	8.84
3.5	5.66	87.68	0.0114	11405	0.1068	106.8	0.0951	95.1	10.51
4.0	8.00	124.00	0.00806	8065	0.0898	89.8	0.0800	80.0	12.50
4.5	11.31	175.36	0.00570	5703	0.0755	75.5	0.0673	67.3	14.87
5.0	16.00	248.00	0.00403	4032	0.0635	63.5	0.0566	56.6	17.68
5.5	22.63	350.73	0.00285	2851	0.0534	53.4	0.0476	47.6	21.02
6.0	32.00	496.00	0.00202	2016	0.0449	44.9	0.0400	40.0	25.00
6.5	45.25	701.45	0.00143	1426	0.0378	37.8	0.0336	33.6	29.73
7.0	64.00	992.00	0.00101	1008	0.0318	31.8	0.0283	28.3	35.36
7.5	90.51	1402.9	0.00071	713	0.0267	26.7	0.0238	23.8	42.04
8.0	128.00	1984.0	0.00050	504	0.0225	22.5	0.0200	20.0	50.00
8.5	181.02	2805.8	0.00036	356	0.0189	18.9	0.0168	16.8	59.46
9.0	256.00	3968.0	0.00025	252	0.0159	15.9	0.0141	14.1	70.71
9.5	362.04	5611.6	0.00018	178	0.0133	13.3	0.0119	11.9	84.09
10.0	512.00	7936.0	0.00013	126	0.0112	11.2	0.0100	10.0	100.0
10.5	724.08	11223.2	0.000089	89.1	0.0094	9.4	0.0084	8.4	118.9
11.0	1024.00	15872.0	0.000063	63.0	0.0079	7.9	0.0071	7.1	141.4
11.5	1448.15	22446.4	0.000045	44.6	0.0067	6.7	0.0060	5.9	168.2
12.0	2048.00	31744.1	0.000032	31.5	0.0056	5.6	0.0050	5.0	200.0
12.5	2896.31	44892.9	0.000022	22.3	0.0047	4.7	0.0042	4.2	237.8
13.0	4096.00	63488.1	0.000016	15.8	0.0040	4.0	0.0035	3.5	282.8
13.5	5792.62	89785.8	0.000011	11.1	0.0033	3.3	0.0030	3.0	336.4
14.0	8192.00	126976.3	0.000008	7.9	0.0028	2.8	0.0025	2.5	400.0

• =

Grade Numbers from etched samples:

9 grains: Grain size #: 5.5

18 grains: Grain size #: 7.5

24 grains: Grain size #: 8.5

15 grains: Grain size #: 7.0



Properties of alloys

Alloy type	Yield Strength (ksi)	Tensile Strength (ksi)	Elongation (%)	Heat Treatment	Specs	Thermal Conductivity (W/mK)
EV31	23.2	45.11	8	Т6	SAE AMS4429A	67.1
WE43	25.2	40.76	13	Т6	SAE AMS4427C	29.7
AZ31	21.8	37	15	T6	ASTM B107	55.5

A T6 heat treat is a 2 step process. The castings are first allowed to cool naturally and are then heated at an elevated temperature in a high temperature oven. After a set period of time the castings are quickly quenched. The castings are then moved to a low temperature oven for the second step of the process.



SPECS

- EV31: SAE AMS4429
 - Magnesium Alloy Castings, Sand 2.8 Nd-1.4Gd-.4Zn-.6Zr (EV31A-T6)
 - Includes information on specifications for composition, casting, cast test specimens (Chemical specimens and tensile specimens), cast corrosion specimens, heat treatment, properties, quality, quality assurance provisions, sampling and testing, reports.
 - https://products.ihserc.com/tmp_stamp/899407550/YANRVEAAAAAAAAAAAAAAA.pdf?sess=899407550&prod=SPECS4
- WE43: AMS4427B
 - Magnesium Alloy Castings, Sand 4.0Y 2.3Nd 0.7Zr (WE43B T6)
 - Includes similar information as SAE AMS4429 accounting for different composition
- AZ31: B107
 - Magnesium-Alloy Extruded Bars, Rods, Profiles, and Wire

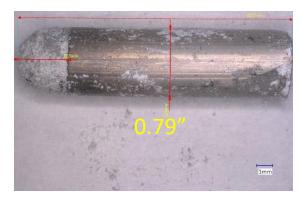


Dimensional Analysis

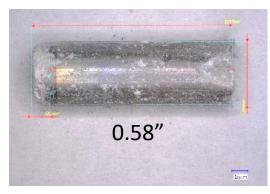


AZ31 3/16" thick rod analyzed using the digital microscope.

Scale as shown.



E 21 3/16" thick rod analyzed using the digital Keyence.
Scale as shown.

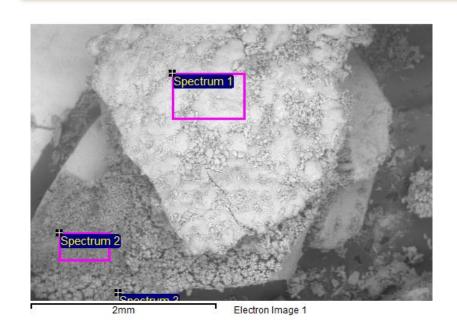


WE43 3/16" thick rod analyzed using the digital Keyence. Scale as shown.

Sample	Length (in)	Diameter (in)	Oxide Cap length (in)
AZ31 (3/16")	0.745	0.185	0.12
EV31 (3/16")	0.79	0.185	0.12
WE43 (3/16")	0.58	0.185	0.04



AZ31 Oxide



pectrum	In stats.	0	Mg	Al	Mn	Zn	Total
Spectrum 1	Yes	43.24	54.98		0.73	1.05	100.00
Spectrum 2	Yes	51.97	43.82	3.81	0.40		100.00
Spectrum 3	Yes	46.33	52.14	0.68	0.86		100.00
Max.		51.97	54.98	3.81	0.86	1.05	
Min.		43.24	43.82	0.68	0.40	1.05	



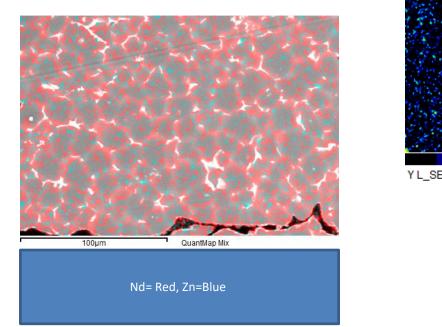
FAA Ban

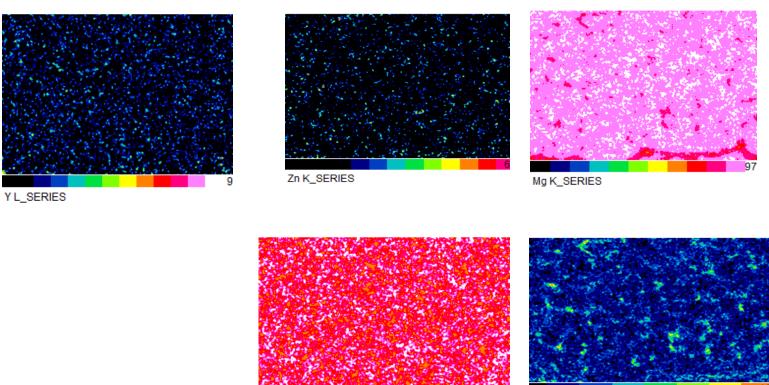
- From Magnesium Elektron.
- "Magnesium Elektron has led an intensive eight-year effort to get to this point with the objective of making modern lightweight magnesium alloys available to aircraft seat designers and manufacturers. Two years ago the Federal Aviation Administration (FAA) allowed the use of certain magnesium alloys under "special conditions," but it has taken until now for the design standard to be formally revised.
- On Aug. 14, the Society of Automotive Engineers (SAE), which develops standards for both the automotive and aviation industries, published SAE AS8049 Revision C, in which a key statement that had previously read "Magnesium alloys shall not be used" was changed to this new wording: "Magnesium alloys may be used in aircraft seat construction provided they are tested to and meet the flammability performance requirements in the FAA Fire Safety Branch document: Aircraft Materials Fire Test Handbook DOT/FAA/AR-00/12, Chapter 25, Oil Burner Flammability Test for Magnesium Alloy Seat Structure."
- Elektron® 43 and Elektron® 21 are the only magnesium alloys that have already met the cited performance requirements by passing extensive flammability tests conducted by the FAA, including seven full-scale aircraft interior tests (for the complete test report, see http://www.fire.tc.faa.gov/pdf/AR11-13.pdf). Developed specifically for demanding aerospace applications, these alloys are high-performance materials that are designed to withstand high temperatures and be resistant to corrosion. Both alloys have proven, long-term performance records, including critical applications in jet engines and military aircraft."
- 21st September 2015



EV31 X-ray dot maps

Gd L_SERIES



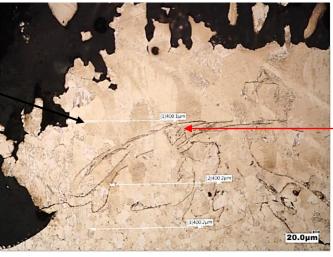


Nd L_SERIES



Etched Samples for 3/16" rod: Comparison of Grains in Inner HAZ

Enlarged transition zone with grain coarsening





Average grain size = 26.7 um

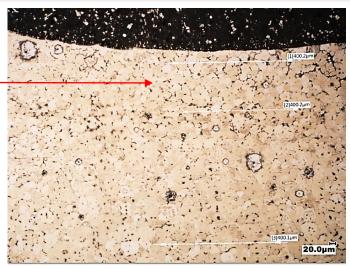
Average grain size = 44.4 um

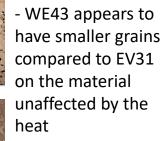
- Particle Counts were taken per ASTM E112 "Standard Test Methods for Determining Average Grain Size" using the line intercept method.
- Analysis was done using ImageJ

Average grain size = 16.6 um

Average grain size = 22.2 um

WE43

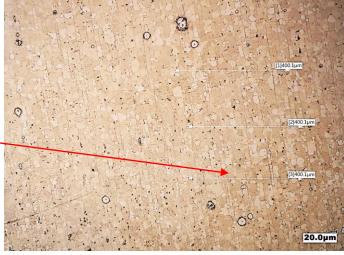




- Grains coarsening

occurs close to HAZ

due to heat effects.



EV31