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A Generic Metallographic Preparation Method for Magnesium Alloys

by James E. Catalano and Laszlo J. Kecskes

ARL-TR-6447 **May 2013**

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)* May 2013	**2. REPORT TYPE** Final	**3. DATES COVERED (From - To)** June 2011–Februrary 2013

4. TITLE AND SUBTITLE
A Generic Metallographic Preparation Method for Magnesium Alloys

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
James E. Catalano and Laszlo J. Kecskes

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
U.S. Army Research Laboratory
ATTN: RDRL-WMM-F
Aberdeen Proving Ground, MD 21005-5069

8. PERFORMING ORGANIZATION REPORT NUMBER
ARL-TR-6447

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

Based on a survey of past metallographic practices and procedures in the literature, a general procedure was developed and evaluated on pure magnesium, a range of currently available high-performance magnesium alloys, and a couple of non-commercial alloys. Our experimental investigation revealed that a single robust composite sequence of polishing and etching steps is possible with slight modifications, based on the alloy composition families tested. The procedure developed shows the microstructural features of the alloys well, delineating grain boundaries and intergranular phases with adequate contrast. Electron microscopy–based observations coupled with energy dispersive x-ray analysis were used to identify the major constituents. Details of the procedure, its limitations, and suggested adjustments or changes are described.

15. SUBJECT TERMS
magnesium, alloys, metallography, polishing, etching, microscopy, composition

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			Laszlo J. Kecskes
Unclassified	Unclassified	Unclassified	UU	52	**19b. TELEPHONE NUMBER** (*Include area code*) 410-306-0811

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

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1. Introduction

There has been renewed interest in the comprehensive exploration of the physical and mechanical properties of magnesium (Mg) and its alloys. A quick survey of recent research publications reveals that these alloys have become an important class of materials for Army applications because of their ultra-low densities, and the vast potential for improvements in properties, especially those capitalizing on their strength-to-weight ratios. Whether these alloys would be used as exterior or interior panels or load-bearing structural components, a range of compositions with widely varying chemistries now exists that could meet the demands of the particular application. Specifically, aside from the traditionally available commercial alloys containing Al, Zn, or Zr, other newly developed Mg alloy families could be used for other, more demanding applications where improvements in thermal stability, corrosion resistance, or flammability are beneficial. These alloys typically include elemental additions of Ca, Y or the rare-earth metals of Nd or Gd.

As in all materials characterization efforts, a proper metallographic preparation of a typical cross section is essential for the correct identification of a particular alloy specimen's substructure and phase chemistry to aid the delineation of the inter-relationship between the major and minor constituent elements and their effect on mechanical properties, such as strength and ductility. There are many preparation recipes intended for different Mg alloys. However, these do not take into consideration the development of newer alloys and the use of alternative alloying additions. As such, the objective of this technical report was to present a survey and revisit existing recipes to develop a single, generic and robust metallographic procedure for the preparation of currently available Mg alloys, regardless of their composition. That is, our primary endeavor was to screen through existing formulations and establish a universal step-by-step procedure for any type of alloy chemistry that, with minor modifications, will reveal the underlying structure, grain morphology, and allow for an ease of phase identification. We relied on the use of scanning electron microscopy (SEM) coupled with energy dispersive x-ray spectroscopy (EDS) to demonstrate the effectiveness of the technique developed.

Table 1 lists most of the alloys acquired for this study. We attempted to obtain alloys that were representative of existing alloy chemistries, both commercial and experimental. In addition to taking the nominal industry designations (see table 2), we have performed an independent verification of the alloys' chemistries and the relative distribution of the constituent elements. In some cases, we did not chemically analyze some of the samples.

Table 1. Mg and its alloys used in this comparative evaluation.

Component (Weight-Percent)	Pure Mg	AZ31B	AZ91C	ZK60	WE43B	AMX602	Elektron 675
Oxygen	<0.0005	<0.0005	<0.0005	0.0009	0.0010	<0.0005	0.0011
Nitrogen	0.001	0.001	0.0020	0.001	0.001	0.001	0.002
Carbon	0.0041	0.0034	0.0010	0.0023	0.0067	0.013	0.0084
Sulfur	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Hydrogen	0.0005	0.0008	0.0016	0.0013	0.0050	0.0252	0.0064
Aluminum	—	3.3	8.8	—	—	5.25	0.011
Manganese	0.0058	0.44	0.25	—	0.010	0.31	—
Zinc	—	1.1	0.66	4.8	0.013	0.018	—
Yttrium	—	—	—	—	4.2	—	Major
Neodymium	—	—	—	—	2.4	—	—
Gadolinium	—	—	—	—	0.63	—	Major
Zirconium	—	—	<0.002	0.46	0.47	—	Major
Silicon	—	0.0039	0.034	—	—	—	—
Copper	<0.0005	<0.0005	0.0023	—	0.0010	—	—
Nickel	0.0010	0.0011	<0.0005	—	0.0039	—	—
Iron	0.042	0.0024	—	—	0.0058	—	0.014
Calcium	—	0.0050	—	—	—	1.87	0.018
Lithium	—	—	—	—	0.0094	—	0.018
Lead	<0.001	—	—	—	—	—	—
Tin	<0.002	—	—	—	—	—	—
Total Others	—	<0.30	<0.30	<0.30	—	—	—
Balance	99.92	Remainder	Remainder	Remainder	Remainder	Remainder	Remainder
Titanium	—	0.027	—	0.012	—	—	—
Lanthanum	—	—	—	—	0.010	—	—
Dysprosium	—	—	—	—	0.35	—	—
Ytterbium	—	—	—	—	0.038	—	—
Cerium	—	—	—	—	0.012	—	—
Silver	—	—	—	—	—	—	—

Table 2. Mg alloying element designations.

Letter	Alloying Element
A	Aluminum
B	Bismuth
C	Copper
D	Cadmium
E	Rare earth
F	Iron
H	Thorium
K	Zirconium
L	Beryllium
M	Manganese
N	Nickel
P	Lead
Q	Silver
R	Chromium
S	Silicon
T	Tin
W	Yttrium
Y	Antimony
Z	Zinc

We selected pure Mg, primarily, to serve as a baseline reference material. The AZ31B and ZK60 alloys were included in the study because they are widely available and already being used commercially. The alloy designations (see table 2) readily reveal that the alloying elements in the AZ series of alloys are Al and Zn, and in the ZK series the corresponding elements are Zn and Zr. In addition to AZ31, we included the AZ61 and AZ91C alloys. The main difference for these alloys is that they have higher Al contents. We also included another alloy, namely WE43B. This alloy, known for its corrosion resistance, contains various levels of rare-earth elements and Y. Of the experimental, nonproduction alloys, reasons for the inclusion of the AMX602 and ZAXE alloys were that, in addition to the more common alloying elements, they contain Ca. Lastly, unlike the other alloys in this set, the Elektron675 is a non-production developmental alloy from Magnesium Elektron North America (MENA), Madison, IL, with a proprietary rare-earth-doped composition.

2. Experimental Procedures

Published literature descriptions of the coarse and fine polishing steps warn about the difficulties related to the preparation of Mg specimens. Because Mg is relatively soft, whether it is unalloyed or with harder second-phase precipitates, the improper preparation, such as the use of an excessive down force, could easily lead to the introduction of artifacts such as reliefs at phase boundaries. Similarly, the specimen could be unintentionally altered creating artificially deformed regions, e.g., inducing deformation twins. Additionally, the descriptions note that the reactivity of Mg with water must be taken into consideration during the preparation of specimens. Thus, it is recommended that the exposure to water is minimized, and possibly the coolant or lubricant is rendered water free.

Our procedure was derived from a review of several different sources, all of which sequentially remove material from the coarse grinding to the final polishing steps. In itself, the identification of the actual polishing sequence does not pose difficulties. It is the application of the etchant in combination with the material removal sequence, which does pose difficulties.

There are several types of preparation recipes in Vander Voort’s text on metallography of materials as well as in the Buehler and Leco material guides (*1*–*3*). These are listed in the appendix.

Table 3 lists a typical sequential polishing procedure for Mg and its alloys. This procedure, obtained from Allied High Tech Products, Inc., was found to be the basis of our procedure. RedLube, Kempad, and FinalA are Allied High Tech Products trade designations. RedLube is a high-viscosity propylene glycol-based lubricant used for soft and/or ductile materials. Kempad is a non-woven, very low-nap textile for use with diamond (1 to 9 µm), designed for good removal and flatness on a wide variety of materials. FinalA is a high-density, non-woven, low-nap, porous polyurethane pad for use with diamond (0.05 to 1 µm), colloidal suspensions, or alumina. According to the product guide, it is “. . . especially effective in eliminating smearing and pullout when preparing soft metals such as copper and aluminum, porous structures/materials, or when preparing materials for SEM or transmission electron microscopy (TEM) evaluation.”[*]

Table 3. Polishing procedure for Mg and its alloys (from Allied High Tech Products, Inc. [*4*]).

	Step	**1**	**2**	**3**	**4**	**5**
Consumables	**Abrasive**	320 Grit	9 µm	3 µm	1 µm	0.04 µm
	Type	SiC	Polycrystalline diamond	Polycrystalline diamond	Polycrystalline diamond	Polycrystalline diamond
	Carrier	Abrasive disk	Suspension	Suspension	Suspension	Suspension
	Polishing Cloth	—	Kempad	Kempad	Kempad	Final A
	Coolant	Water	RedLube	RedLube	RedLube	—
Settings	**Platen Speed/Direction**	300 RPM comp	150 RPM contra	150 RPM contra	150 RPM contra	250 RPM contra
	Head Speed	150 RPM	150 RPM	150 RPM	150 RPM	150 RPM
	Force (lbf/mount)	5	5	5	5	3
	Time	Until flat	3 min	3 min	5 min	1 min

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[*] Quote comes from the Allied High Tech Products, Inc. Product Catalog. http://www.alliedhightech.com/products/download.html (accessed April 30, 2013).

This procedure includes further notes, as follows:

1. Mg dust is a fire hazard. Never cut or grind Mg dry.
2. SiC paper should be coated with a thin layer of wax (e.g., candle wax, paraffin) to prevent particle embedding. Apply a piece of wax to the center of the SiC disc while it is spinning at about 250 RPM with water as lubricant. Move the wax outward slowly until the entire disc is coated uniformly with wax.
3. The sample should be cleaned with anhydrous denatured alcohol in between polishing steps (Steps 2 to 4) to prevent oxidation. First, rinse the polished surface with alcohol and then immerse the sample into alcohol bath to wash away any residuals on the side. Avoid cleaning the sample with brush or cotton swab/puff, which will scratch the polished surface.
4. Cleaning colloidal silica after the last step without water could be challenging. It was found that holding the sample under running water for a second and then rubbing the polished surface quickly with the thumb did not appear to damage the sample. Pure Mg is less sensitive to water than its alloy and can withstand the water rinse a bit longer.
5. Allied RedLube contains mainly propylene glycol and methyl alcohol, which can be used as a water-free polishing lubricant. A small amount of water in the Allied propylene glycol-based diamond suspensions did not appear to harm the water sensitive samples.
6. Pure Mg is softer than its alloy and some areas on the sample might be smeared as a result. Adding about 5 to 10 volume % of hydrogen peroxide (30% concentration) into the colloidal silica suspension will help define the grains.
7. To prevent the polished surface from oxidizing in the air during storage, seal the sample with a small piece of polyimide tape that has a silicone based adhesive and will not leave residue on the surface.
8. Mg is a soft metal and it can easily have deformed grains and smeared layers on the polished surface. For best results, alternate light etching and light re-polishing a few times until the desired outcome is reached.

Our preparation apparatus entails the use of an Allied MetPrep 4 Grinder/Polishing system coupled with the AD-5 Fluid dispenser system (Allied High Tech, Rancho Dominguez, CA). This system comprises a polishing wheel and specimen holding fixture that could be contra or complementarily rotated relative to the polishing wheel (or platen). The apparatus allows for the adjustment of platen speed, specimen rotation speed and direction, cycle time, lubricating fluid selection, sample down force, and water rinse. The use of the AD-5 fluid dispenser facilitates unattended operations wherein the polishing suspensions or lubricants could be applied automatically. It may be noted that we have used products from different metallographic suppliers, whichever fit best, for our grinding and polishing needs.

All specimens were mounted in 1 1/4-in-diameter Isofast hot mounting medium (Struers, Westlake, OH). This mounting compound is a diallylphthalate resin with glass fibers; it was deemed the most suitable for all of the different alloy specimens.

After a series of preliminary trial-and-error attempts in developing a unified sequence, each alloy sample was sequentially prepared using a five-step procedure. The steps are listed here.

Step 1–Coarse grinding: using 320-grit SiC paper coated with a thin layer of wax, with a platen speed of 300 RPM, specimen holding fixture rotating in a complementary direction to the platen with a head speed of 150 RPM, water coolant, and 5 lb of down-force pressure were used until the specimen was flat.

Step 2–Fine grinding: once flat, using 9-µm MetaDi supreme polycrystalline diamond suspension and Trident pad, both from (Buehler, a division of Illinois Tool Works, Inc., Lake Bluff, IL), the specimens were fine ground to a 9-µm finish. For this step, the platen speed was 150 RPM, the specimen holder was contra rotated at 150-RPM head speed, using a MetaDi fluid extender, 5 lb of down-force pressure for 5 min.

Step 3–Coarse polishing: using 3-µm MetaDi supreme polycrystalline diamond suspension, Trident pad, platen speed of 150 RPM, specimen holder contra rotating and head speed 150 RPM, MetaDi fluid extender, 5 lb of down-force pressure, 5 min.

Step 4–Fine polishing: using 1-µm MetaDi Supreme polycrystalline diamond suspension, Trident pad, platen speed 150 RPM, specimen holder contra rotating and head speed 150 RPM, MetaDi fluid extender, 5lb of down-force pressure, 5 min.

Step 5–Final polishing: using 0.04-µm Purple colloidal silica suspension (Allied), FinalA pad (Allied), platen speed 300 RPM, specimen holder complementarily rotating and head speed 150 RPM, 3 lb of down-force pressure, 2 min.

Once the polishing sequence was completed, the samples were then quickly rinsed in water, and immediately submerged in ethyl alcohol, followed by 10 min of ultrasonic cleaning. After the metallographic preparation was completed, the samples were kept in an evacuated belljar.

The MetaDi diamond suspension, Trident, and MetaDi fluid extender are all Buehler trade names, whereas, the Purple colloidal silica suspension and FinalA cloth are Allied trade names. The Trident polishing cloth is claimed to be versatile, and it is designed for intermediate polishing before the final polishing step. It is suitable for hard and soft specimens. The MetaDi fluid extender is used to keep the diamond suspension ‘suspended’ longer; it consists of a mixture of polyglycol and water. The Purple colloidal silica suspension is basic (pH greater than 7), non-stick, and rinseable. It is intended for final polishing. The color is only for identification purposes from other lubricants.

It may be noted that small amounts of water in the Buehler propylene glycol-based diamond suspension/extender did not appear to harm the Mg samples.

All specimens were etched using a 100-ml ethanol, 10-ml distilled water, 10-ml acetic acid, and 5-g picric acid etchant. Immersed and using gentle agitation 5–20 s. Though not precisely the same, the etchant used in our effort is closest to Vander Voort's etchant no. 10. However, an examination of Vander Voort's other etchants, i.e., nos. 10–13, as well as Leco's etchant no. 125 (*1, 3*) indicate a similar reliance on varying ratios of acetic and picric acids.

Microscopic evaluation of the samples was performed using a Hitachi S4700 field-effect, cold-cathode SEM (Hitachi North America, Gaithersburg, MD), equipped with an EDS analyzer (EDAX Inc., Mahwah, NJ). The SEM was operated at 20keV in either secondary electron mixed detector mode (SE[M]) or using an external annulus-type Yttrium-aluminum-garnet (YAG) backscatter detector to collect a purely backscattered electron image (BEI) (identified as YAGBSE). It may be noted that in Mixed mode, one of the two secondary electron detectors is located in the pole piece of this instrument and it will impart the image with a backscatter component. Thus, in addition to a secondary electron image (SEI), the resultant overall image will contain a superimposed BEI as well. Qualitative analysis was used to identify constituent elements. In turn, to determine the relative ratios of the elements identified, a standardless semi-qualitative analysis routine was used. It turned out that prior to examination, occasionally some of the samples had to be repolished and re-etched. That is, sometimes storage in partial vacuum was insufficient to prevent the onset of oxidation.

3. Results

Table 1 lists the Mg alloys used in this comparative evaluation. As shown in the table, there is some variation in composition from alloy to alloy. However, most of the alloying elements are present in relatively low concentrations. As such, they would be expected to be present mostly as grain boundary phases.

3.1 Pure Mg, Production and Commercially Available Alloys

3.1.1 Pure Mg

By far, compared to other alloys, the surface characteristics of pure Mg was found to be most sensitive to the length of time between etching and SEM examination. As shown in figure 1, the nascent Mg surface is mostly obscured by a buildup of an oxide layer. In fact, in this case, this oxide layer is sufficiently thick and has a distinct enough morphology by itself to be easily misinterpreted as the actual microstructure.

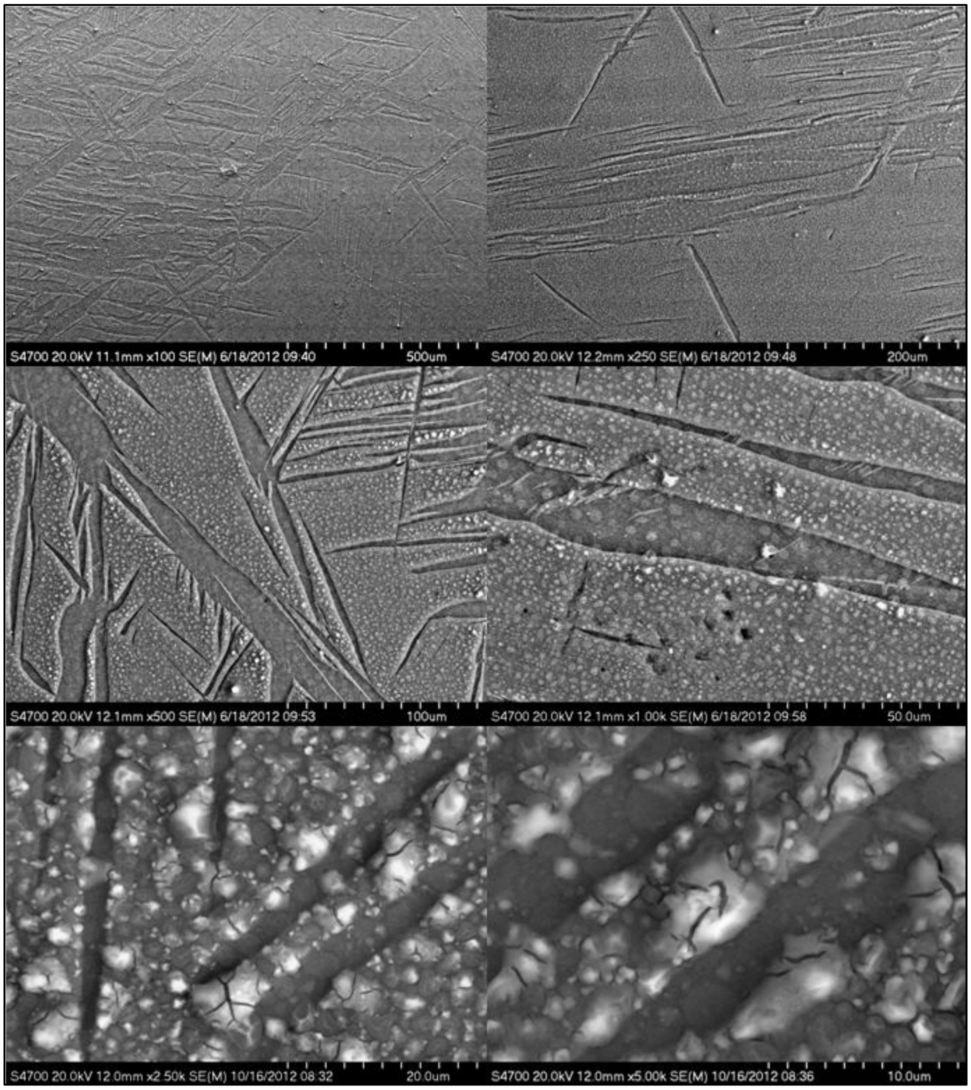

Figure 1. The post-etched surface of pure Mg reveals what appears to be a partitioned, interpenetrating microstructure. A closer examination reveals blistering and buildup on the surface, which has a high concentration of oxygen, as analyzed by EDS, e.g., $Mg_{97.2}O_{2.8}$ and $Mg_{97.7}O_{2.3}$. To the inexperienced metallographer, this could be easily misinterpreted as part of the real microstructure.

3.1.2 Pure Mg Repolished and Re-Etched

The pure Mg sample was repolished and immediately examined with SEM. The pure BEI micrographs show that the underlying structure is hard to see, however, there is some electron density variation, resembling some type of grain structure. As shown in figure 2, this grain structure shows mostly lenticular grains. However, the oxidation obscured structure seen before, is not present. EDS only shows Mg.

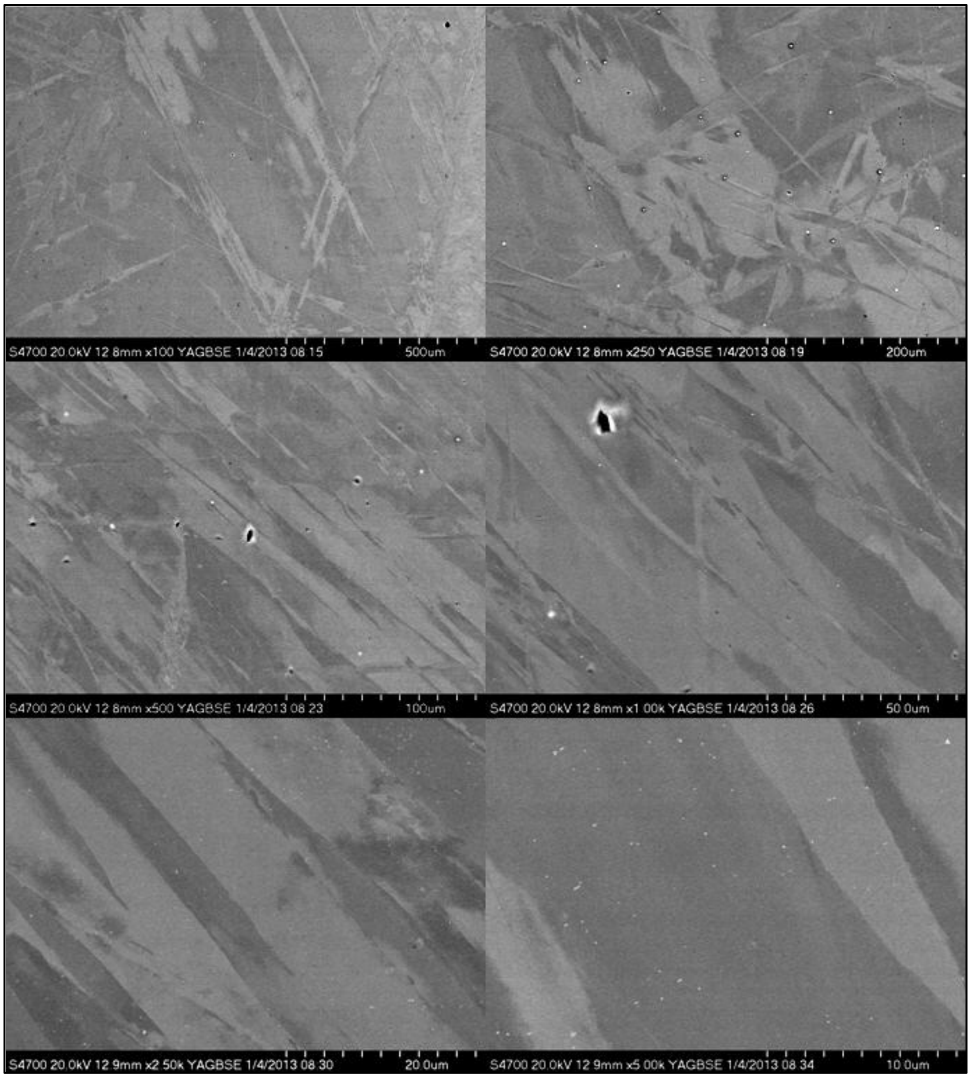

Figure 2. The post-repolished, but unetched surface of pure Mg barely reveals its true grain structure. The weak contrast between grains, probably attributed to slight differences in electron channeling from each grain, is inadequate to accurately assess the microstructure.

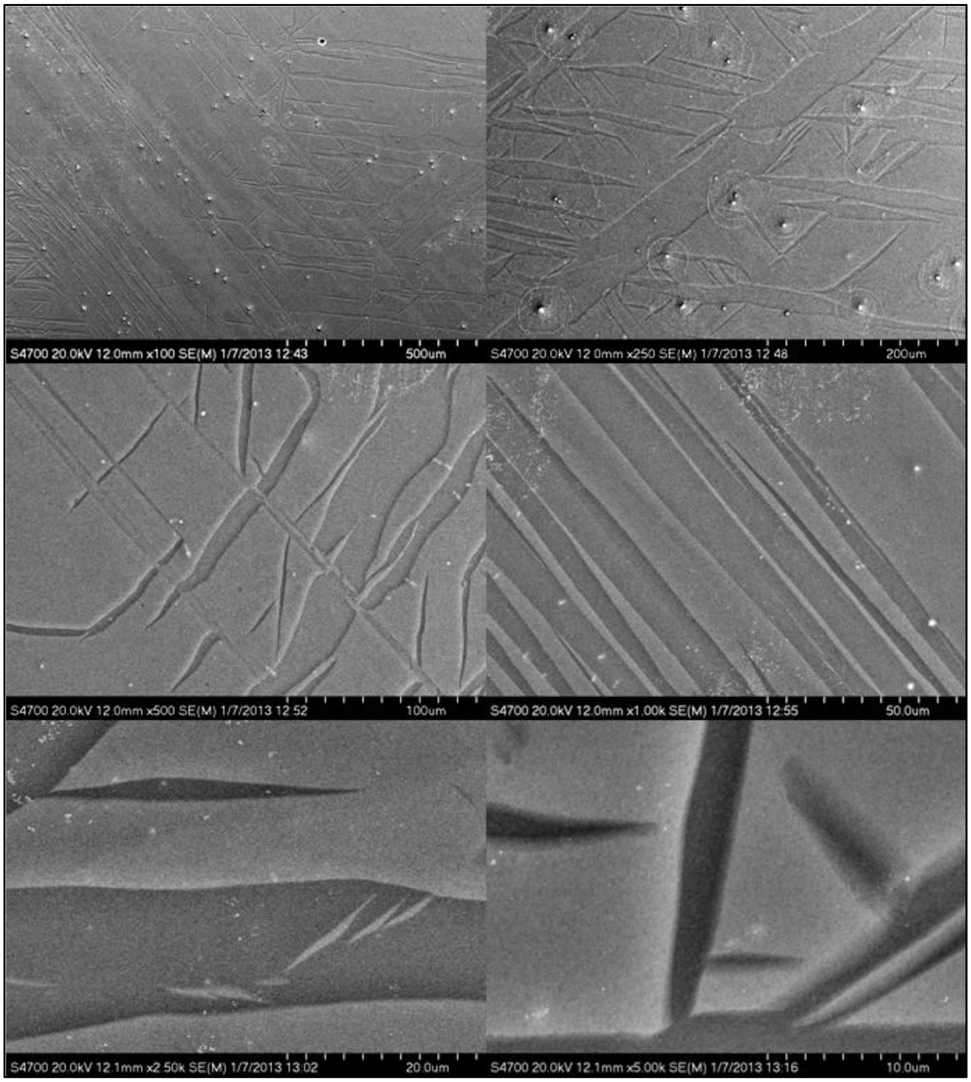

Figure 3. The carefully re-etched surface of pure Mg clearly reveals its true grain structure. There is adequate contrast between grains to accurately assess the microstructure. In this case, there is no blistering or oxide buildup on the surface. The grains of this sample consist of a series of stacks of layered parallel, lenticular grain colonies (perhaps twins?). (Interestingly, this underlying structure was somewhat already apparent in the overetched, oxidized condition. That is, possibly oxidation is grain orientation dependent.)

3.1.3 AZ31 Alloy

Figures 4–6 display SEMs of the three AZ31 specimens. Recall, the SEM images were taken using the secondary electron detectors of the instrument, however, in the mixed mode, SE(M). In mixed mode, the image will not only incorporate the traditional surface features visible to, but also will contain atomic number contrast, usually associated with backscattered electrons.

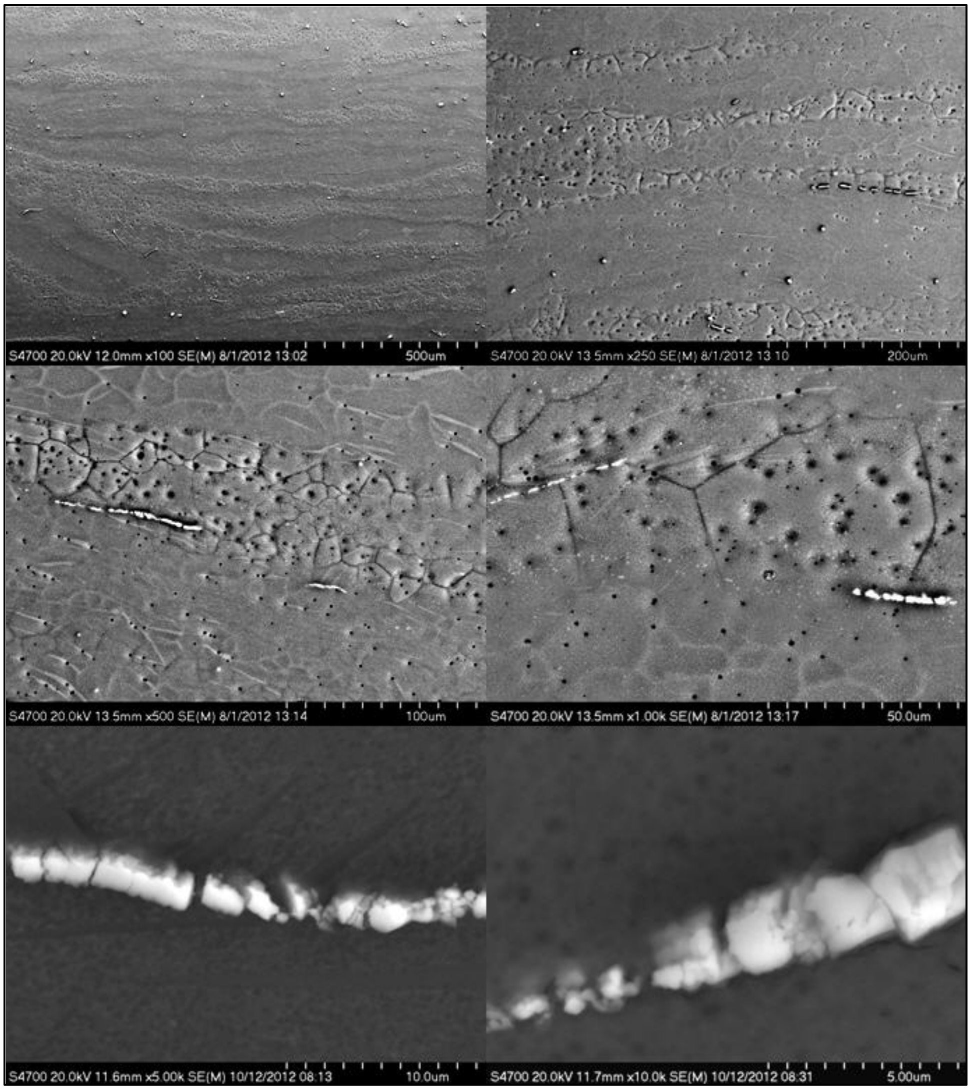

Figure 4. In this sample, the AZ31 alloy reveals a banded microstructure, with alternating bands of coarse and fine grains. The dark spots are etch pits. The overall, average composition, as determined with EDS is $Mg_{972}Al_{2.1}Zn_{0.7}$. Another EDS spot analysis showed $Mg_{95.8}Al_{3.0}Zn_{1.16}$. The anticipated Mn from the chemical analysis is below the EDS detection level. It is in the electron-dense ‘stringers’ where the Mn mostly resides; the relative ratio of the elements in these precipitate structures is $Mg_{46.1}Al_{41.8}Mn_{11.8}Zn_{0.3}$.

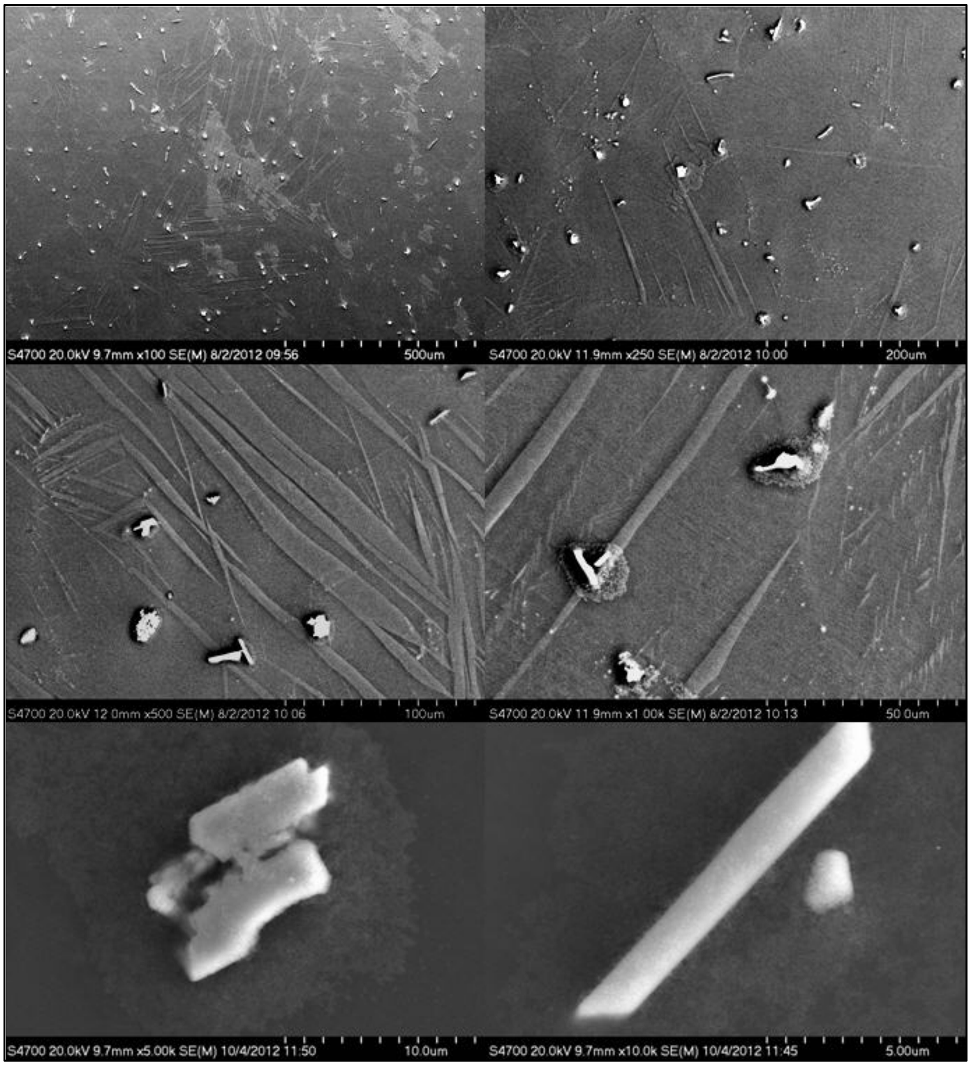

Figure 5. Unlike the first sample, in the second sample of AZ31B alloy, a more uniform, equiaxed and isotropic microstructure presents itself, with lath-like precipitates within the grains. The overall, average composition, as determined with EDS is $Mg_{97.6}Al_{2.4}$. (Zn was not detected.) The lighter lath-like phase consists of $Mg_{98.3}Al_{0.9}Zn_{0.8}$. The Mn-containing platelet and rod-like precipitates are dispersed and isolated; the ratio of the elements in these precipitate structures is $Mg_{33.8}Al_{31.7}Mn_{32.5}Si_{2.0}$.

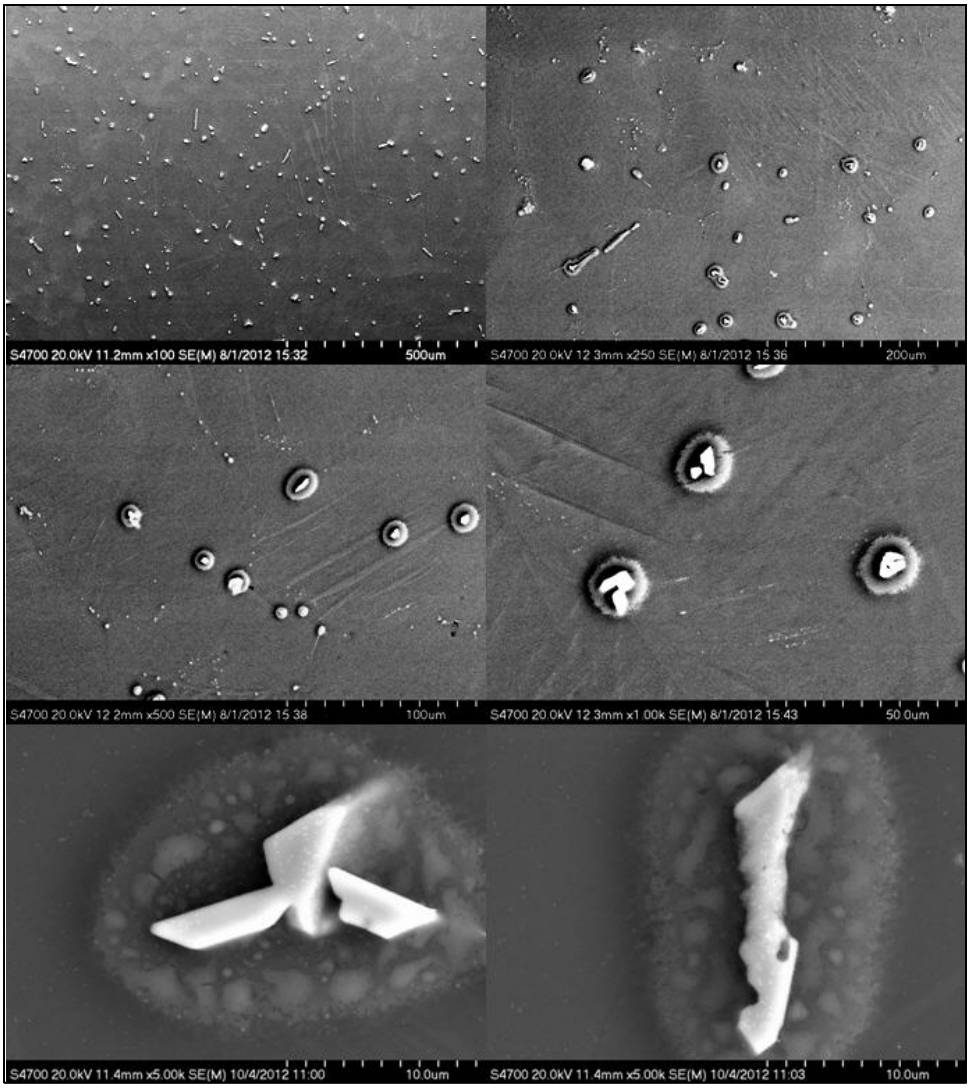

Figure 6. In the third sample, the second AZ31B alloy sample reveals a similar microstructure as that observed in the first AZ31B alloy sample, with alternating bands of coarse and fine grains. The overall, average composition, as determined with EDS is $Mg_{96.5}Al_{2.6}Zn_{0.9}$. The fine laths are $Mg_{98.1}Al_{1.3}Zn_{0.6}$. Again, the Mn, identified in the chemical analysis, is below the EDS detection level. It is found in the more electron-dense precipitates where the Mn mostly resides; the relative ratio of the elements in these precipitate structures is $Mg_{28.8}Al_{30.2}Mn_{41.0}$. Note that there is staining surrounding the precipitates; this is more pronounced than those around the precipitates in the other AZ31B samples.

As it is conveyed in the images from the three samples, this alloy is actually not a single phase, but instead contains precipitates within the grains and, in some cases, along the grain boundaries. It is interesting to note that while, in general, the EDS analysis was consistent with the overall chemical analysis of the samples, most of the alloying elements were tied up in the precipitates. The metallographic polishing sequence combined with the etching clearly reveals the sub and fine structures in the alloy. In addition to the grain-to-grain boundary contrast evidenced in the micrographs, the edges of the precipitates are well defined. There is little or no smearing. Note, in the second and third samples, there is some staining around the precipitates. This is attributed to artifacts associated with the quality of etchant. However, overall, the preparation methodology is quite suitable for this alloy.

3.1.4 AZ61 Alloy

There was only one AZ61 specimen. As the images in figure 7 reveal, the structure is quite different from that of the previous AZ31 sample. This alloy is much finer grained. Unfortunately, this alloy was not chemically analyzed. Based on the nominal composition, it was expected to contain about twice the Al level than that in the AZ31 alloy. Since EDS analysis indicated about the same Al level in the grains, it is speculated that the additional levels of Al is located along the grain boundary phase. EDS analysis results indicate $Mg_{97.5}Al_{2.5}$ for the grain and $Mg_{18.8}Al_{32.2}Mn_{45.3}Si_{1.1}O_{2.6}$ along the boundaries. As was observed before, the Al and Mn are mostly contained along the grain boundaries. The appearance of the blistering and oxygen is an indication of contamination from either the mounting or polishing media, or the sample was overetched.

3.1.5 AZ61 Alloy Re-Etched

Because of the presence of oxygen, the apparent “cellular” structure of the AZ61 specimen is highly suspicious. This structure actually is an artifact of poor timing of the metallographic preparation and SEM examination. In particular, the time lapse between the metallographic preparation and subsequent electron microscopy is critical for Mg alloys. During storage, an extended time can result in rapid buildup of an oxide layer, which can easily obscure the true microstructure. It could also be likely that some regions (e.g., grain boundaries) are more sensitive to oxidation; as such, the oxide buildup is more rapid there. As the images in figure 8 reveal, the structure is quite different from what it appeared before. The overetching and resultant blistering completely obscures the actual microstructure. The grain size and morphology are fairly uniform and equiaxed. However, in addition to the fine grains there is a dispersion of generally much larger spherical particles.

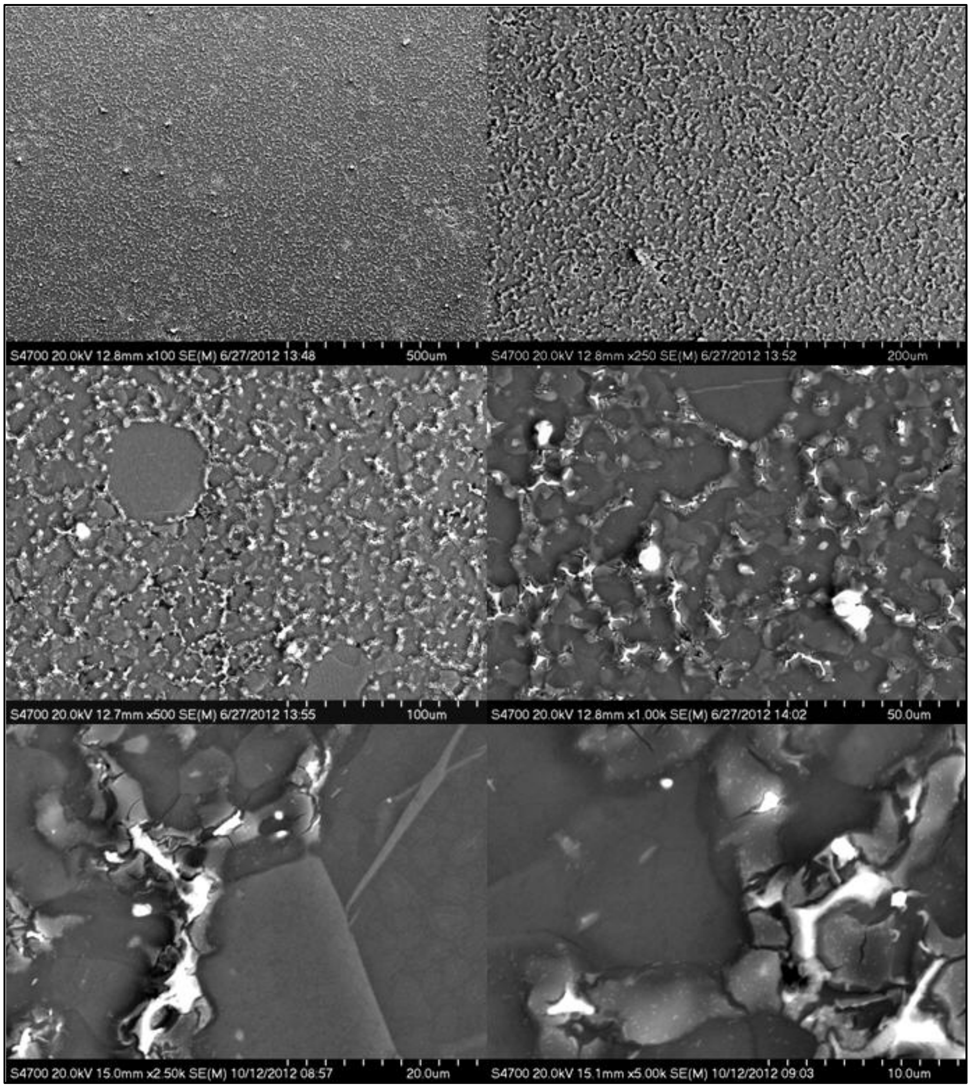

Figure 7. The fine-grained AZ61 alloy reveals a microstructure, with darker equiaxed grains with a grain boundary-like phase. The darker grain composition, as determined with EDS, is $Mg_{97.5}Al_{2.5}$. The string of grain-like blisters, appearing as a “cellular” structure, contains slightly more Al, $Mg_{94.4}Al_{5.6}$. Upon further examination, some of the very bright grains in this phase contain $Mg_{18.8}Al_{32.2}Mn_{45.3}Si_{1.1}O_{2.6}$. The source of the oxygen and Si are most likely contamination.

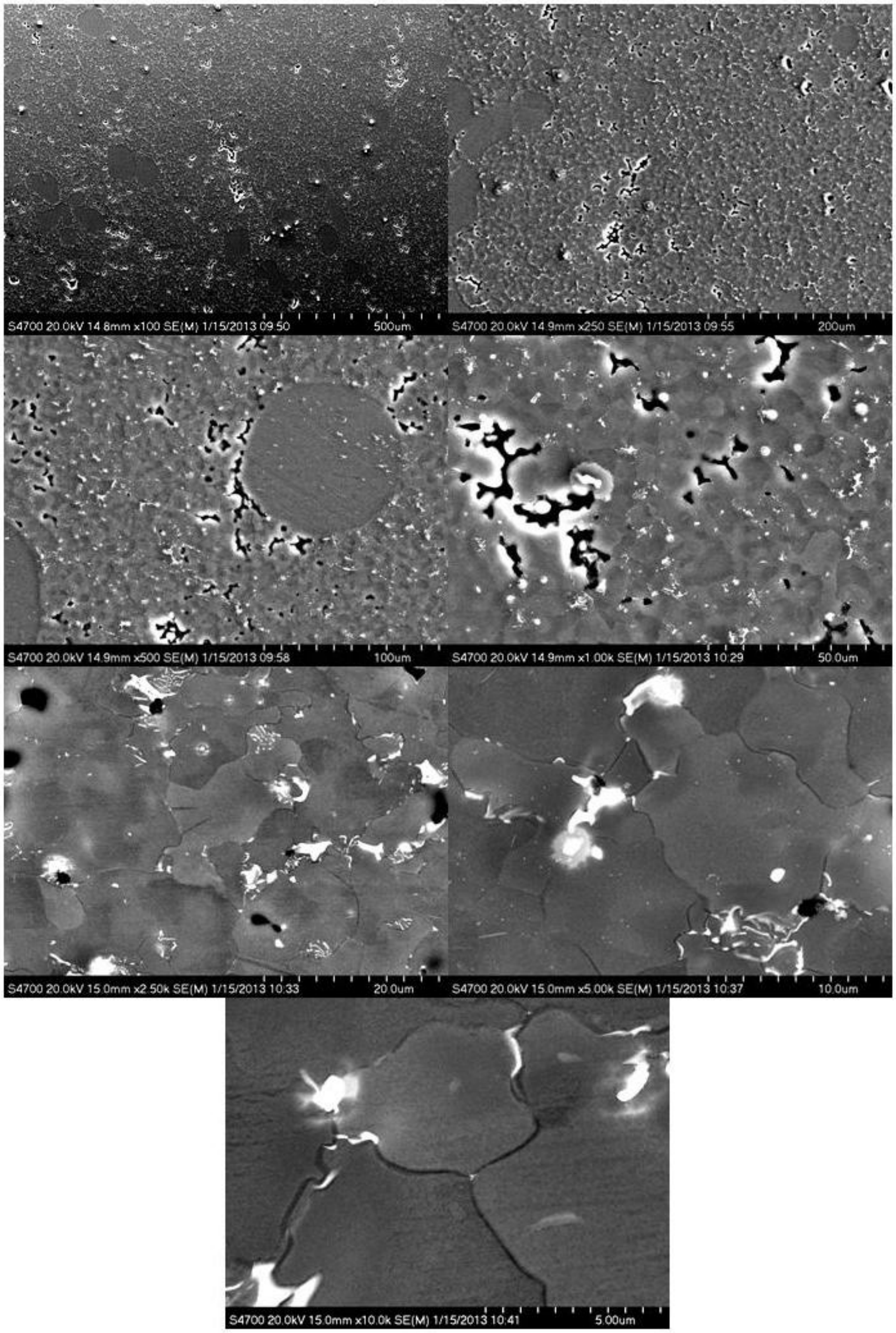

Figure 8. The repolished and re-etched fine-grained AZ61 alloy no longer reveals an easily incorrectly interpretable "cellular" microstructure. Instead, it consists of fine, equiaxed grains with a lighter intergrain phase and grain boundary precipitates. The darker grain composition, as determined with EDS, is $Mg_{98.4}Al_{1.6}$. The interspersed string of lighter grains contain slightly more Al, $Mg_{87.5}Al_{12.5}$. The very bright precipitates in the grain boundaries contains $Mg_{26.2}Al_{42.7}Mn_{31.1}$. In some locations these appear to have eutectic-like features.

3.1.6 AZ91 Alloy

There was only one AZ91 specimen. As the images in figure 9 reveal, the structure is quite different from those of the previous AZ31 or AZ61 samples. This alloy is much finer grained. Based on the nominal composition, it was expected to contain about three times the Al level than that in the AZ31 alloy. EDS analysis confirms the chemical analysis results. EDS analysis results indicate $Mg_{92.4}Al_{7.6}$ for the lighter grains. The grain boundary phase was not analyzed, but from the atomic number contrast, it is expected that it would contain less Al. The large bright acicular particles contain mostly Al and Mn, with an overall composition of $Mg_{2.7}Al_{47.5}Mn_{47.9}Si_{1.9}$. It may be noted that the apparent grains actually have a eutectic-like structure with finely dispersed dendrites.

3.1.7 ZK60A Alloy

The ZK60A alloy composition is representative of another class of Mg alloys because it contains the alloying elements of Zn and Zr. Aside from the large-grained cobble stone appearance, the structure and morphology are unremarkable. The actual grains are probably larger, containing several precipitates, barely discernible after noting the faint black lines, appearing in the micrographs. As shown in figure 10, EDS analysis reveals that the majority of Zn is contained in the precipitates, whereas the darker phase has a lower average atomic number density. The uniformly dispersed, very fine lighter grey colored marks are probably etching stains or residue.

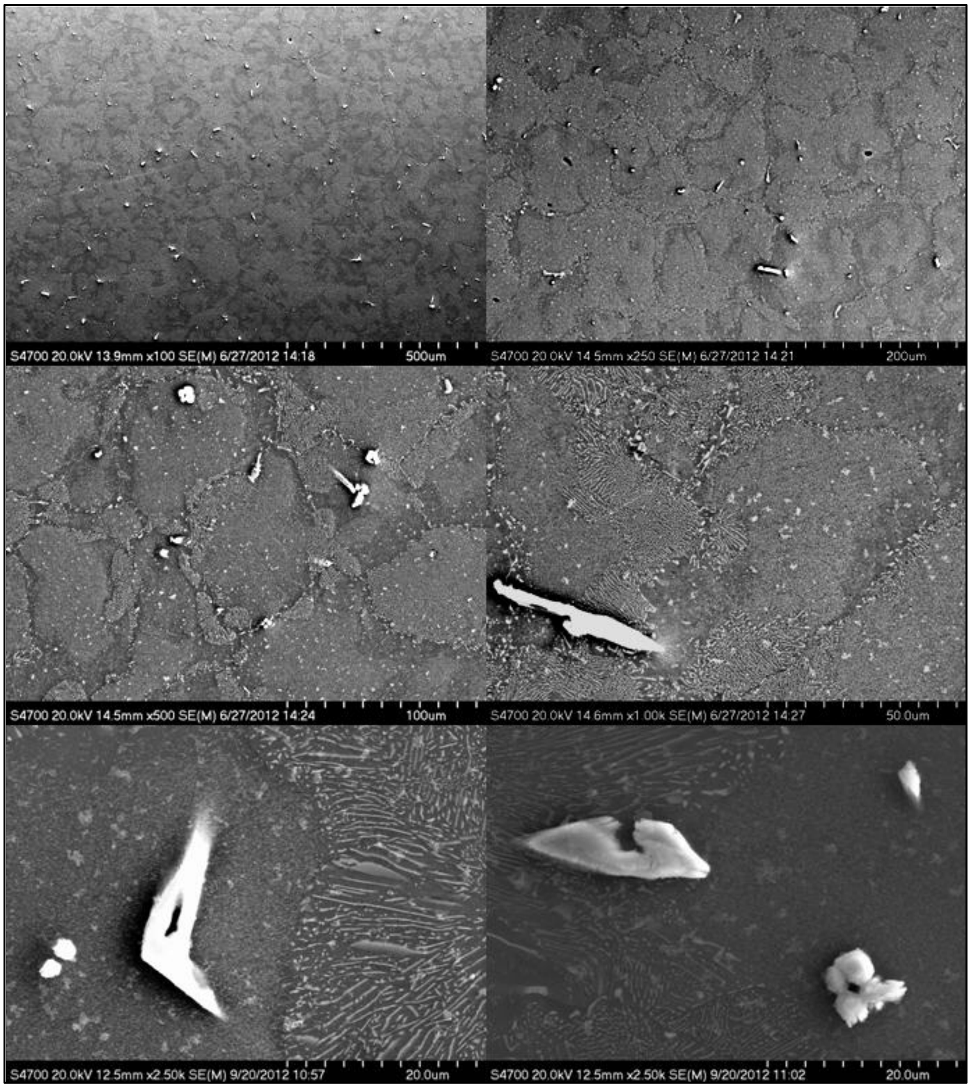

Figure 9. The considerably more fine-grained AZ91 alloy reveals a partitioned two-phase microstructure with lighter equiaxed grains with a darker grain boundary phase. The lighter grain composition, as determined with EDS, is $Mg_{90.5}Al_{6.7}Zn_{0.6}Si_{2.2}$. Examination at higher magnifications indicates that these equiaxed grains have a dendritic, eutectic-like, fine structure. The grain boundary phase is $Mg_{92.4}Al_{7.6}$. Additionally, the larger precipitates are devoid of Mg, but instead consist mostly of Al and Mn, $Mg_{2.7}Al_{47.5}Mn_{47.9}Si_{1.9}$.

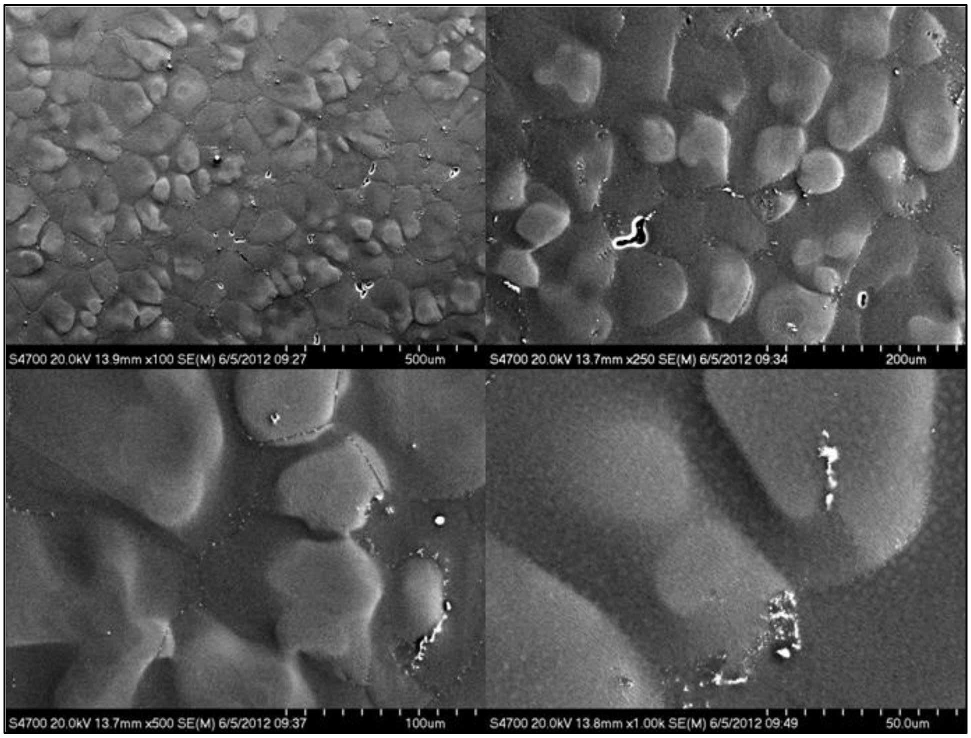

Figure 10. The much coarser-grained ZK60A alloy reveals a ‘cobble-stone-like’ partitioned two-phase microstructure with lighter and darker grain-like features. Noting, the presence of a network of black demarcation lines, the actual grains are probably much larger, containing several precipitates. The lighter grain-like precipitates, as determined with EDS, consist of Mg96.5Zn3.5. The darker grain-boundary-like phase is Mg97.2Zn2.8. The random, much smaller white precipitates contain Mg66.5Zn24.6Zr8.9.

3.1.8 WE43 Alloy

The WE43 alloy composition is representative of a different class of Mg alloys; it contains small quantities of the rare-earth alloying elements of Nd and Gd. In addition, as revealed in table 1, it contains larger quantities of Y and Zr. As shown in figure 11, the grains tend to contain $Mg_{95.8}Nd_{0.8}Y_{3.4}$. In contrast, the bright fine precipitates are more rich in Zr; specifically, $Mg_{54.8}Gd_{0.3}Y_{3.1}Zr_{32.5}O_{9.3}$. In addition to the large randomly bright particles, there is a uniformly dispersed ultrafine nodular precipitates.

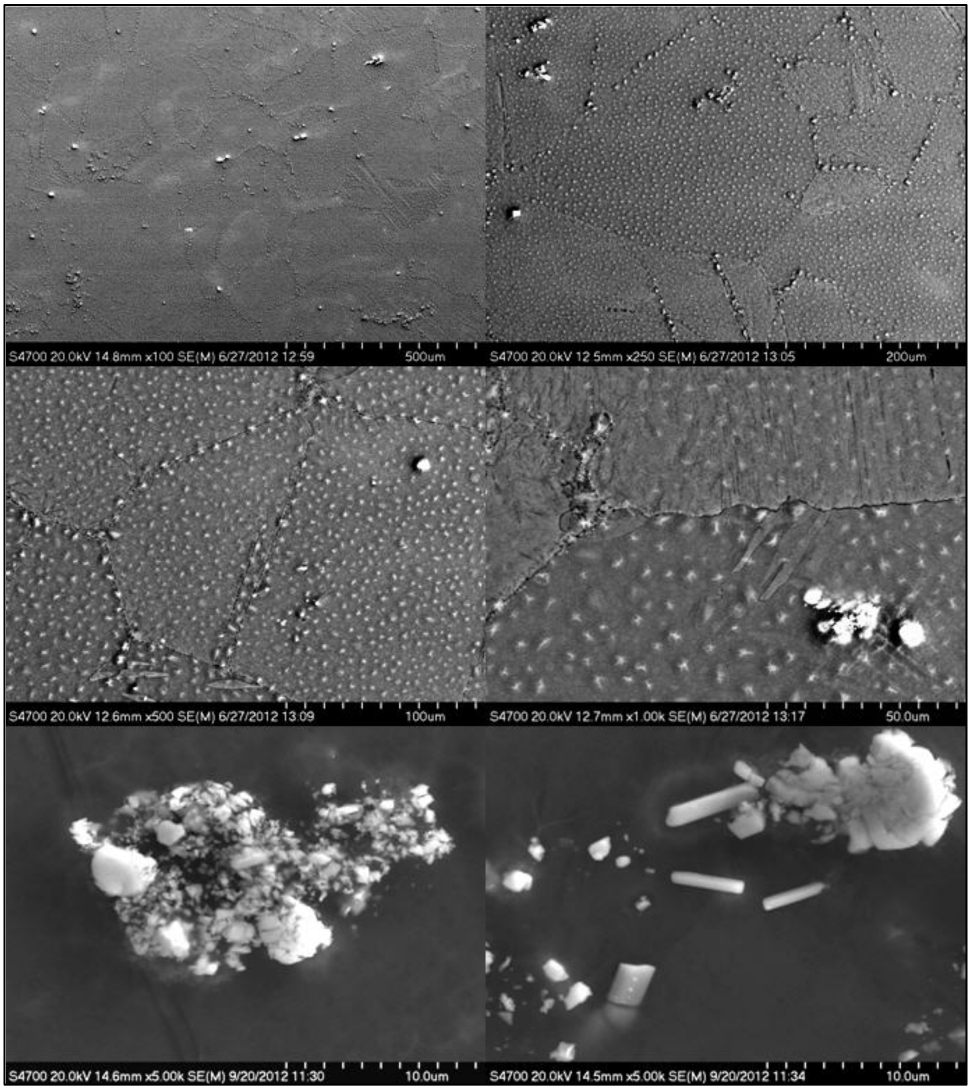

Figure 11. The coarse-grained WE43 alloy reveals very large grains with finely dispersed clusters of precipitates. EDS analysis of the equiaxed grains reveals $Mg_{96.5}Nd_{0.8}Y_{3.4}$ and $Mg_{96.3}Nd_{0.8}Y_{3.0}$. The lighter grain composition, as determined with EDS, is $Mg_{96.5}Zn_{3.5}$. The lighter, fine precipitate clusters contain $Mg_{54.8}Gd_{0.3}Y_{3.1}Zr_{32.5}O_{9.3}$.

3.1.9 WE43 Alloy Re-Etched

The first examination of WE43 alloy sample indicated a surface layer of fine nodular precipitates. Upon repolishing and re-etching the larger precipitates are still present; however, the fine precipitates are absent. Instead, this alloy consists of very large heavily deformed grains, interspersed with extremely fine precipitates. As shown in figure 12, the grains tend to contain $Mg_{96.2}Nd_{0.7}Y_{3.1}$. Whereas, the bright intergrain precipitates are highly rich in Zr, $Mg_{57.4}Gd_{0.3}Y_{2.9}Zr_{39.3}$. Note that some of the intergrain precipitates are surrounded by etching residue. Note, the extensive deformation indicated by the striated appearance of the grains.

3.2 Experimental and Non-Production Alloys

3.2.1 AMX602 and ZAXE1711 Alloys

The AMX and ZAXE alloys are special Ca-containing alloys, which were produced by a special spinning water atomization process followed by an extrusion step. Microstructural and mechanical properties of these alloys have been described elsewhere; see references 5 and 6. As such, the alloy's microstructure was expected to be extremely fine-grained, highly oriented, and acicular.

Repolishing and re-etching the ZAXE alloy did not significantly improve one's ability to discern the microstructure. This is probably attributable to the ultrafine grained nature of the microstructure or perhaps the fine dispersion of precipitates. EDS analysis of the grains revealed $Mg_{92.4}Al_{5.8}Ca_{1.0}Zn_{0.9}$ and $Mg_{81.7}Al_{6.0}Ca_{1.9}$ $Zn_{1.0}O_{9.4}$.

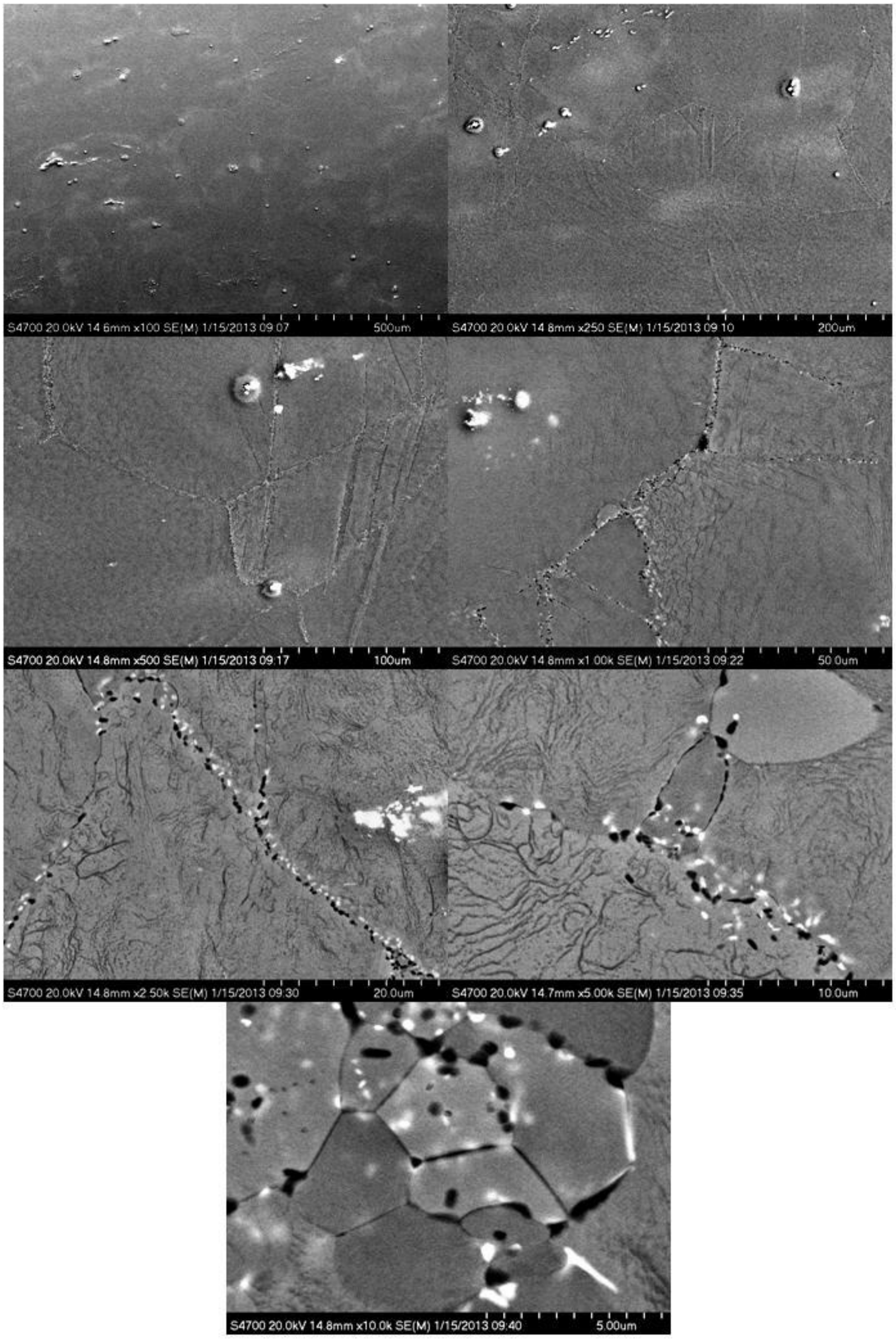

Figure 12. The repolished and re-etched coarse-grained WE43 alloy reveals very large grains with finely dispersed clusters of precipitates along the grain boundaries. The larger grains actually consist of much finer grains. EDS analysis shows $Mg_{96.2}Nd_{0.7}Y_{3.1}$ for the grains and $Mg_{57.4}Gd_{0.3}Y_{2.9}Zr_{39.3}$ for the bright precipitates.

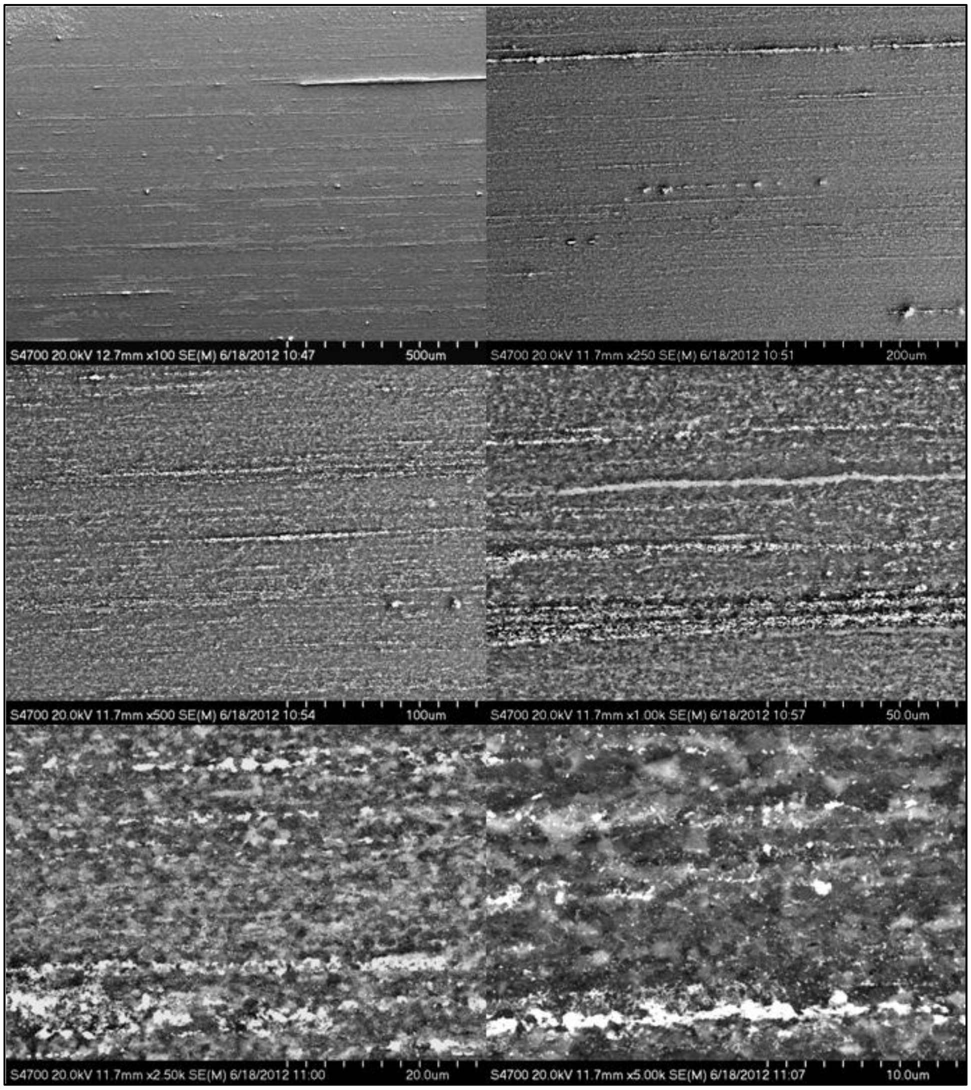

Figure 13. The extremely fine-grained AMX602 alloy contains a typical microstructure characteristic of an extruded material. The darker mottled region consists of mostly $Mg_{93.6}Al_{4.6}Ca_{1.9}$ and the bright striated regions are Al-rich, $Mg_{55.7}Al_{42.8}Ca_{1.6}$.

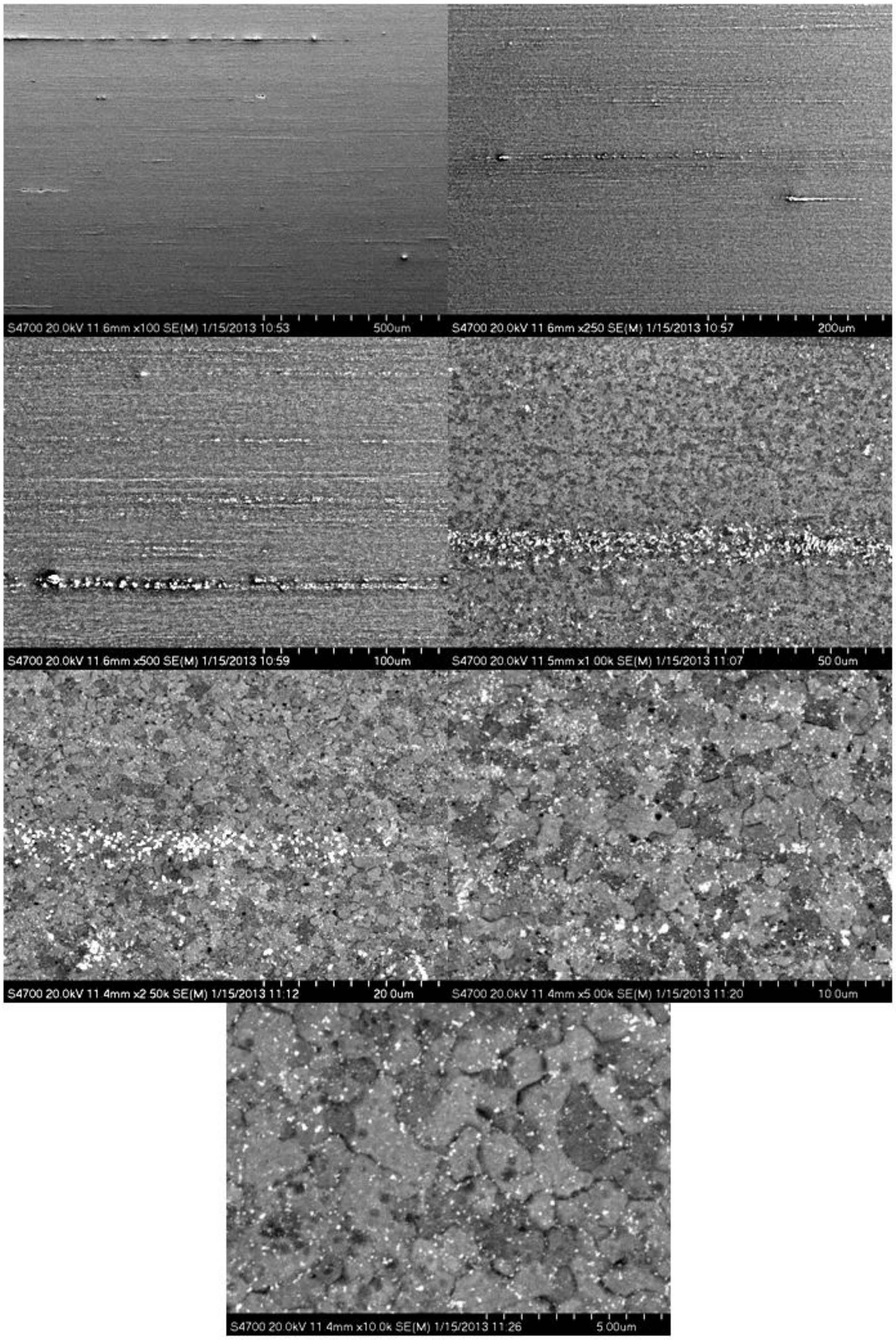

Figure 14. Re-etching of the extremely fine-grained AMX602 alloy confirmed the overall microstructure. The darker mottled region consists of mostly $Mg_{93.7}Al_{4.4}Ca_{1.8}$ with equiaxed grains. The striated regions, consisting of submicrometer precipitates are Al- and Ca-rich, $Mg_{55.7}Al_{25.98}Ca_{9.9}O_{8.4}$. The presence of oxygen in the EDS spectrum is indicative of excessive oxidation, perhaps from overetching.

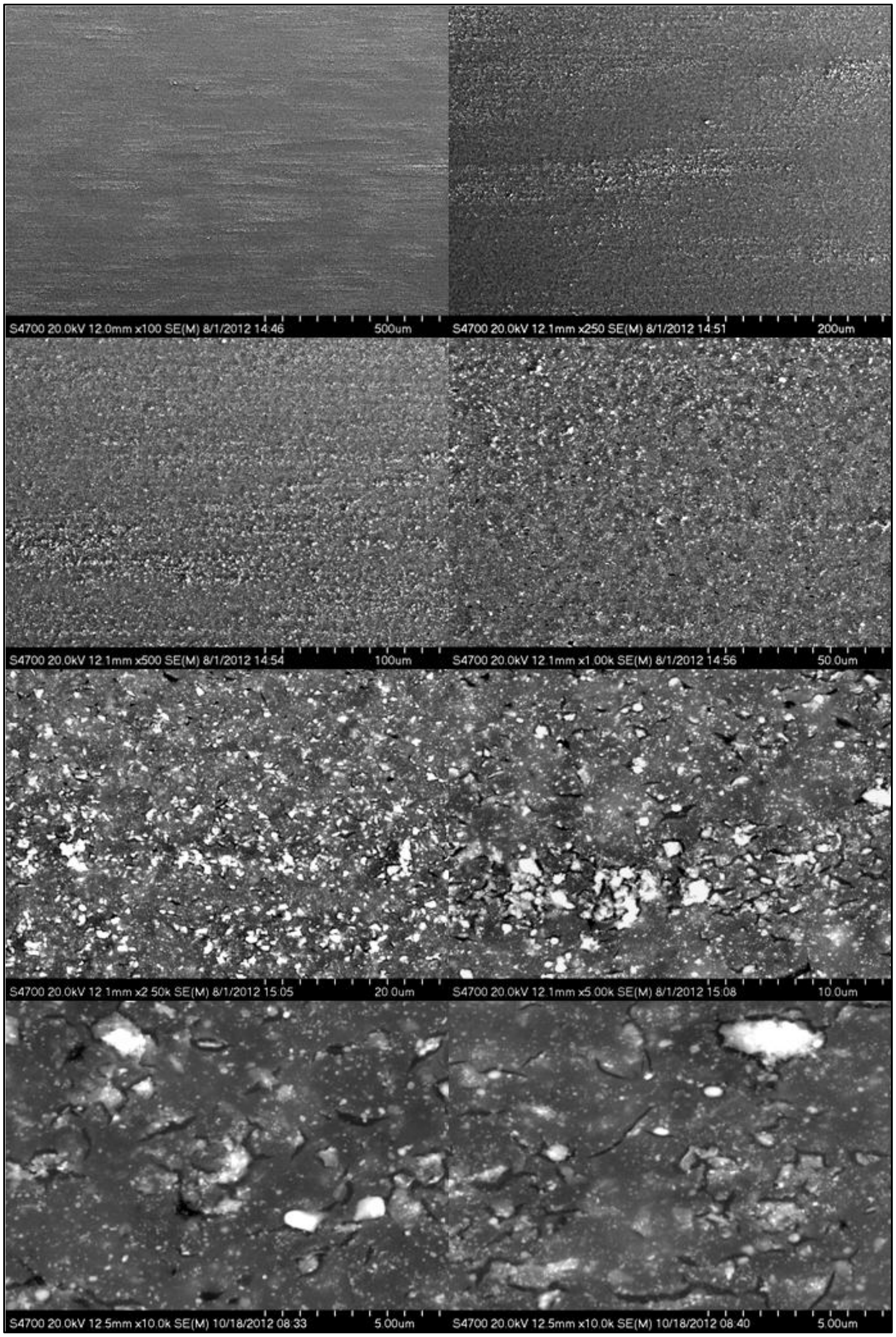

Figure 15. Similar to the AMX602 alloy, the fine-grained ZAXE1711 alloy is also extruded. The darker phase consists of $Mg_{84.6}Al_{6.5}Ca_{1.0}Zn_{0.8}O_{7.1}$, whereas the light particles are more Ca- and oxygen-rich, consisting of $Mg_{81.8}Al_{6.6}Ca_{1.4}Zn_{0.7}O_{9.5}$.

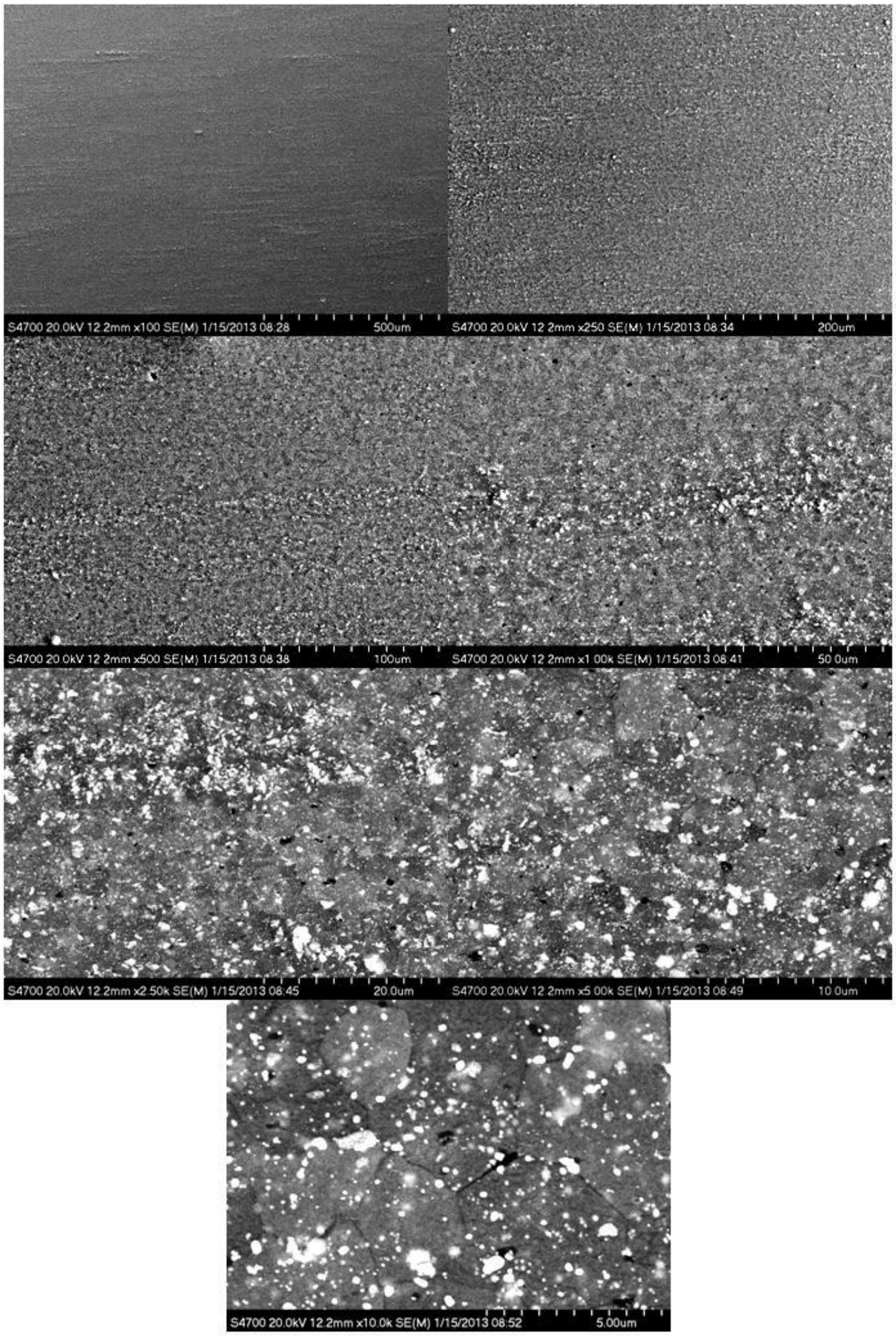

Figure 16. After re-etching, the fine-grained ZAXE1711 alloy reveals an equiaxed grain microstructure. No EDS was performed on this alloy.

3.2.2 Elektron675 Alloy

The last alloy we included in this limited-scope study was the Elektron675 alloy, conditionally available from MENA (*7*). The etching process does not leave any artifacts on the surface and, as seen in figure 17, the elemental contrast is good to excellent without saturation or other detrimental effects during microscopy.

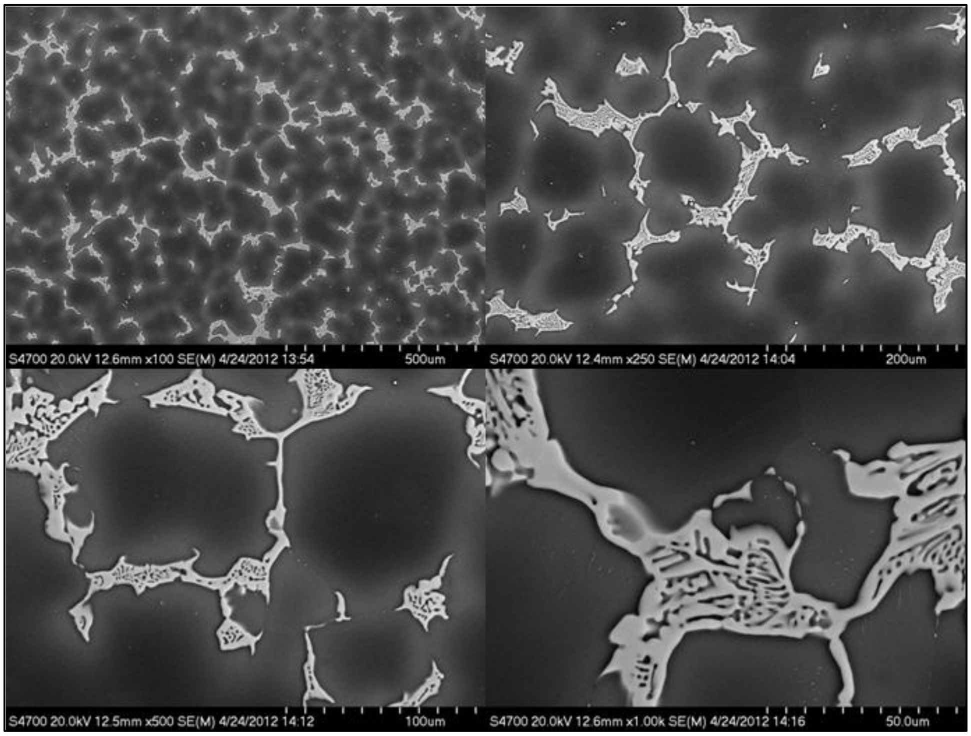

Figure 17. The very coarse grained MENA Elektron675 is an alloy with a proprietary composition, still in the experimental development stage. The dark grey regions consists of $Mg_{96.6}Gd_{1.0}Y_{2.4}$ while the light grain boundary phase is more rich in Gd, $Mg_{82.5}Gd_{8.1}Y_{9.4}$.

4. Discussion

Our comparative etching study demonstrated that for a range of Mg alloy chemistries, a single polishing/etching procedure can be applied to get a good first glance of their microstructures. However, not all aspects of the overall procedure are as robust, flexible, and forgiving as anticipated. That is, while the polishing sequence is quite forgiving and produces an excellent surface finish, the etching step is found only to be adequate for the range of compositions examined. Moreover, use of the method presupposes a prior familiarity with metallography techniques, skill level, or intuitive feel, so that a condition of overetching is recognized early. Specifically, it was rather difficult to obtain a clean and good surface finish and discernible grain-to-grain contrast, especially in pure Mg. Similarly, because of the variability of the time delay between etching and microscopic examination, the final polish and etching step had to be repeated several times to remove residual oxide buildup from the surface, which otherwise obscured the surface. For consistent results this delay time had to be kept to a minimum. Lastly, it may be noted that certain alloying additions, especially the rare-earth elements, significantly improved stability and allowed for greater latitude and flexibility in the etching response.

While the composition did affect the alloy's response to the etchant, further aspects of the alloy microstructure such as grain size, morphology, number and type of precipitates, necessitated other adjustments to the procedure to obtain optimum final polish and etching results. The periphery around precipitate structures, grain boundaries, and any other discontinuity led to oxide residue buildup. Obviously, some of the more corrosion resistant alloys were better in this respect, but partial staining was always present. In other cases, due to the increasing strength of the etchant, likely caused by evaporation of the alcohol solvent, it was necessary that some of the samples had to be re-etched and exposure time limited to accurately reveal grain boundaries.

5. Conclusions

A series of polishing and etching procedures was identified in literature sources for Mg and its alloys. A composite approach was then developed, with the intent to be robust and without the need for considerable adjustments as determined by alloy composition. The procedure was evaluated on pure Mg, and Mg-Al-Zn-, Mg-Zn-Zr-, or Mg-rare-earth-based alloys. Additionally, a few experimental alloys rich in other alloying elements, e.g., with Ca or Gd, were examined. The overall quality of the procedure was found to be adequate, only necessitating minor adjustments in the application time of the etching solution. Typical micrographs of the alloys display well the underlying microstructures with an easy identification of the various phases present. Major adjustments are probably unnecessary; perhaps all that is needed is adjustment to stabilize the longevity of the etching solution.

6. References

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3. Johnson, C. A. *Metallography Principles and Procedures*; Leco Corporation: St. Joseph, MI, 1992; p 38, pp 46–47.
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5. Kondoh, K.; Hamada, E. A.; Imai, H.; Umeda, J. *Microstructures and Mechanical Responses of Powder Metallurgy Noncombustive Magnesium Extruded Alloy by Rapid Solidification Process in Mass Production*; ARL-CR-647; U.S. Army Research Laboratory: Aberdeen Proving Ground, MD, May 2010.
6. Ayman, E.; Kondoh, K.; Imai, H.; Umeda, J. *Effect of Different La Additions on the Microstructure and Mechanical Properties of Hot Extruded SWAP Mg-Al-Zn-Ca Powder*; Transactions of the Joining and Welding Research Institute: Osaka University, Osaka, Japan, 2009; Vol. 38, No. 1.
7. DeLorme, R. D. Magnesium Elektron North America, Madison, IL. Private communication, January 9, 2013.

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Appendix. Published Procedures and Etchants for Mg and Its Alloys

Table A-1. Etchant compositions for revealing microstructure of Mg and its alloys. From *Metallography, Principles and Practice*.[1]

Etchant: Composition	**Comments**
1: 25-ml water 75-ml ethylene glycol 1-ml HNO_3	Glycol etch, general-purpose etch for pure Mg and alloys. Swab sample 3–5 s for F and T6 temper alloys, 1–2 min for T4 and O temper alloys.
2: 19-ml water 60-ml ethylene glycol 20-ml acetic acid 1-ml HNO_3	Acetic-glycol etch for pure Mg and alloys. Swab sample 1–3 s for F and T6 temper alloys, 10 s for T4 and O temper alloys. Reveals grain boundaries in solution-treated castings and most wrought alloys.
3: 100-ml alcohol 1010-ml HNO_3	Nital, for pure Mg and alloys. Immerse sample for up to about 60 s.
4: 90-ml water 10-ml HF	For pure Mg and some alloys. Immerse sample for 3–30 s, agitate gently. Darkens $Mg_{17}Al_{12}$, leaves $Mg_2Al_2Zn_3$ unetched.
5: Solution a: 100-ml ethanol 5-g picric acid Solution b: 90-ml water	Use after etch No.4 to darken matrix for increased contrast. Use fresh. Use 10 ml of solution a plus solution b, immerse sample 15–30 s.
6: 10-ml water 100-ml ethanol 5-g picric acid	For Mg and alloys. Use fresh. Immerse sample for 15–30 s. Produces grain contrast.
7: 100-ml water 100-ml ethanol 5-g picric acid	For Mg-Si alloys. Good contrast between Mg_2Si and Mn, Mg_2Si blue, Mn dull gray. Immerse sample for up to 30 s.
8: 100-ml alcohol 0.7-ml H_3PO_4 5-g picric acid	Phospho-picral etch, for pure Mg and some alloys. Composition is critical. Use fresh, immerse sample 1–30 s, agitate gently. Used to estimate amount of massive $Mg_{17}Al_{12}$ compound in heat-treated castings or wrought alloys. Stains solid solution, leaves compound white.
9: 100-ml water 0.2–2-g oxalic acid	For pure Mg and most alloys. Swab sample 6–10 s.
10: 95-ml alcohol 10-ml acetic acid 5-g picric acid	For Mg-Al and Mg-Al-Zn alloys. Immerse sample 15–60 s, agitate gently. Use fresh. Sometimes reveals grain boundaries.
11: 10-ml water 100-ml ethanol 5-ml acetic acid 5-g picric acid	For Mg and alloys. Reveals grain boundaries.

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[1] Vander Voort, G. F. *Metallography, Principles and Practice*; McGraw-Hill, Inc.: New York, NY, 1984; pp 658–660.

Table A-1. Etchant compositions for revealing microstructure of Mg and its alloys. From *Metallography, Principles and Practice* (continued).

Etchant: Composition	**Comments**
12: 15-ml water 100-ml alcohol 30-ml acetic acid 6-g picric acid	For Mg-Al, Mg-Al-Zn, and Mg-Zn-Zr alloys. Immerse sample 1–30 s, agitate gently. Use fresh.
13: 10-ml water 50-ml ethanol 16–20 ml acetic acid 3-g picric acid	For Mg and alloys. Reveals trace of basal plane. Sensitive to aging temperature. Use fresh, immerse sample 15 s. Forms amorphous film. When dry, film cracks parallel to the basal plane. Reveals compositional variations within grains.
14: 95-ml water 5-ml acetic acid	For Mg-Al alloys up to 6% Al. Swab sample 3–5 s, also used for pure Mg.
15: 90-ml water 2–10 tartaric acid	For Mg-Al (to 6% Al), Mg-Mn, and Mg-Mn-Al-Zn alloys. Sometimes reveals grain structure in castings. Immerse sample 5–15 s. For higher Al content, use 20-g tartaric acid.
16: 100-ml water 2–11-g citric acid	For Mg, Mg-Cu, and Mg die-casting alloys. Immerse sample about 30 s.
17: 100-ml water 5–10-g citric acid	For Mg-Mn wrought alloys and solution-treated castings. Reveals grain boundaries. Swab sample 5–30 s.
18: 85-ml water 15-ml HNO_3 12-CrO_3	For Mg-Al alloys. Sometimes produces grain contrast in heat-treated castings. Immerse sample 10–30 s. Increase water content for high-Al alloys.
19: 85-ml ethylene glycol 15-ml HNO_3	For alloys of Mg, 1% Zn, and 0.6% Zr. Immerse sample 2 s.
20: 1 part HNO_3 1 part water saturated with sodium fluorosilicate and potassium tartarate	For Mg-U alloys.
21: 95.5-ml water 2.5-ml HNO_3 1.5-ml HCl 0.5-ml HF	For Mg-Zn alloys. Mix 1-ml etch with 100-ml water. After 10-s etch, Mg_7Zn_3 attacked, MgZn relatively unattacked.
22: 1000-ml water 50-g CrO_3 4-g Na_2SO_4	For Mg-Zn alloys. After 2-s etch, MgZn severely attacked, Mg_7Zn_3 slightly attacked.

Table A-1. Etchant compositions for revealing microstructure of Mg and its alloys. From *Metallography, Principles and Practice* (continued).

Etchant: Composition	**Comments**
23: 2 parts water 1 part H_3PO_4 10 parts 6% picral	Polarized-light etchant for Mg. Sharp extinctions when basal plane is parallel or perpendicular to plane of polish.
24: 100-ml water	For Mg and wrought alloys. Immerse sample 10–30 s.
25: 100-ml water 10-g ammonium persulfate	For wrought Mg alloys. Produces grain contrast. Swab sample until surface is brown.
26: 100-ml ethanol 2-ml HCl	For Mg and alloys, immerse sample 10 s.
27: 3000-ml ethanol 25-ml HNO_3 15-ml HCl	For Mg and alloys, immerse 1–5 s.
Electrolytic Etchants	
28: 100-ml water 10-g NaOH	For Mg alloys. Use at 4 V DCdc. Cu cathode, 2–4 min. Etch immediately after polishing.
29: 20-ml water 20-ml ethanol 40-ml H_3PO_4	For Mg and many alloys. Use at 10–35 V dc, Mg cathode, 1–10 min.
30: 100-ml water 10-ml HF	Color etch for Mg and alloys. Use stainless steel cathode, 20 °C, 0.6–0.9 V dc, 10–15 mA/dm2, 20–30 min, moderate stirring. Remove sample with current on.

Table A-2. Macroetchant compositions for Mg and its alloys. From *Metallography, Principles and Practice*.[2]

Etchant: Composition	**Material**	**Comments**
1: 75–95-ml water 5–25-ml acetic acid	ZK60A	Immerse sample in solution at room temperature until desired contrast is obtained. Alloys with zinc may form smut on surface which can be removed by dip in a 1:1 solution of HF and H_2O. Reveals flow lines.
2: 90-ml water 10-g tartaric acid	Mg and Mg alloys	Immerse sample in solution at room temperature for up to about 3 min. Reveals flow lines.
3: 80-ml water 20-ml acetic acid 5-g $NaNO_3$	AZ31B AZ61A AZ80A	Immerse sample in solution at room temperature for 1–5 min. Reveals flow lines.
4: 10-ml water 5-ml acetic acid 100-ml alcohol 5-g picric acid	AZ61A AZ80A	Immerse sample in solution at room temperature for up to 3 min. Use finely ground surface. Reveals grain size and flow lines.
5: 10-ml water 10-ml acetic acid 70-ml alcohol 3.5-g (or 6 g) picric acid	AZ21 AZ31	Immerse sample in solution at room temperature for up to 3 min. Use finely ground surface. Reveals grain size and flow lines.
6: 20-ml water 5-ml (or 20-ml) acetic acid 50-ml alcohol 2.5-g (or 6-g) picric acid	ZK60A	Immerse sample in solution at room temperature for up to 3 min. Use finely ground surface. Reveals grain size and flow lines.
7: 80–90-ml water 10–20-ml HNO_3	Mg-Al-Zn-Mn (C-alloy)	Immerse sample in solution at room temperature for up to 5 min. Remove smut with a 1:1 solution of HF and H_2O. Reveals flow lines and internal defects.
8: 24-ml water 75-ml ethylene glycol 1-ml HNO_3	Mg-Al-Zn-Mn (C-alloy)	Glycol etch. Reveals grain structure. Immerse sample in solution at room temperature until desired contrast is obtained.
9: 19-ml water 60-ml ethylene glycol 20-ml acetic acid 1-ml HNO_3	All Mg alloys	Immerse sample in solution at room temperature until desired contrast is obtained. Reveals grain structure.
10: 1000-ml water 180-ml HNO_3 180-g $Na_2Cr_2O_7$	Mg alloys	Immerse sample in solution at room temperature until desired contrast is obtained. General purpose etch for revealing defects.

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[2] Vander Voort, G. F. *Metallography, Principles and Practice*; McGraw-Hill, Inc.: New York, NY, 1984; pp 523–524.

Table A-2. Macroetchant compositions for Mg and its alloys. From *Metallography, Principles and Practice* (continued).

Etchant: Composition	**Material**	**Comments**
11: 1000-ml water 25-ml HNO_3 10-ml HF 280-g CrO_3	Mg alloys	Immerse sample in solution at room temperature until desired contrast is obtained. Reveals abnormal grain growth and surface defects.
12: 100-ml alcohol 0.7-ml H_3PO_4 4-g picric acid	Mg alloys	Immerse sample in solution at room temperature repeatedly to obtain desired contrast by staining. Reveals segregation of intermetallic compounds and cracking. Use finely ground surface.
13: 100-ml water 2-10-g ammonium persulfate	Mg alloys	Immerse sample in solution at room temperature until desired contrast is obtained. Reveals flow lines.
14: 100-ml ethanol saturated with picric acid 10-ml acetic acid	Mg-Al-Zn-Mn alloys	Use fresh solution to reveal grain structure.
15: 85-ml water 15-ml HNO_3 12-g CrO_3	Mg alloys	Immerse sample in etch. Reveals grain size.
16: 1000-ml water 24-g sodium acid phosphate 4-g potassium ferricyanide	Mg alloys	Use finely ground specimen. Immerse specimen in solution at room temperature for 40 min to 2 h. Reveals flow lines.

Table A-3. Electrolytic solutions for Mg and its alloys. From *Metallography, Principles and Practice*.[3]

Etchant: Composition	Current Density	Voltage, DC	Temperature, °C	Time, min	Comments
1: 625-ml ethanol 375-ml H_3PO_4	4.5–5 A/dm^2	1–3	Room Temperature	10	Current density drops to 0.5 A/dm2 as polishing progresses. When an anode film forms, wash it off quickly to prevent pitting. Inadequate for polarized light studies.
2: 10-ml HCl 90-ml cellosolve	2 A/dm^2	10–15	20–30	1–2	After initial polarization, the voltage can be reduced to 5 V with 1 A/dm2 current density. Good for polarized light studies.
3: 200 or 250-ml water 380-ml ethanol 400-ml H_3PO_4	0.2 A/cm^2	10	20–50	2	Use Mg cathode. Remove sample with current on. If a film forms during washing, remove with dilute HNO3 swab.
4: 800-ml ethanol 80-ml bytyl cellosolve 160-g sodium thiocyanate	1.3 A/cm^2			10 s	Dissolve sodium thiocyanate in ethanol, add butyl cellosolve last.
5: 10-ml water 100-ml ethanol 10-ml butoxyethanol 20-g sodium thiocyanate 10-g EDTA (Na+ salt form)		23	20		After eletropolishing, wash in alcohol jet.
6: 700-ml acetic acid 300-ml perchloric acid	1.5 A/dm^2	20–30	<30	1–2	Use stainless steel or nickel cathode.

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[3] Vander Voort, G. F. *Metallography, Principles and Practice*; McGraw-Hill, Inc.: New York, NY, 1984; p 582.

Table A-4. Buehler metallography. Four-step procedure for magnesium (Mg) alloys.[4]

Surface	Abrasive/Size	Load lb (N) /Specimen	Base Speed (RPM)/Direction	Time (min:s)
CarbiMet 2 Abrasive discs (waterproof paper)	220- to 320- (P240 to P400) grit SiC[a] Water cooled	5 (22)	200–250 Comp.[b]	Until plane
TexMet pad[c]	9-µm MetaDi oil-based diamond slurry	5 (22)	120–150 Contra[d]	6:00
TexMet pad	3-µm MetaDi oil-based diamond slurry	5 (22)	120–150 Contra	5:00
TexMet pad	1-µm MetaDi oil-based diamond slurry	5 (22)	120–150 Contra	4:00
ChemoMet cloth	~0.05-µm MasterPolish or MaterPrep alumina	6 (26)	120–150 Contra	1:30–3:00

[a] SiC surfaces were coated with wax to minimize embedment.
[b] Comp. = complementary (platen and specimen holder both rotate in the same direction).
[c] The TexMet pad is a medium hard non-woven pad for all types of material preparation from pre- to final polishing stages with a diamond from 15 to 3 µm. The ChemoMet is a porous, soft micro-nap pad, made from a synthetic material, intended for general use where chemo-mechanical polishing is needed.
[d] Contra = platen and specimen holder rotate in opposite directions.

Table A-5. Buehler metallography. Commonly used etchants for metals and alloys. Mg and alloys.[4]

Composition	Comments
25-ml water 75-ml 2–5 ethylene glycol 1-ml HNO_3	Glycol etch, general purpose etch for pure Mg and alloys. Swab specimen seconds for F and T6 temper alloys; 1–2 min for T4 and temper alloys.
19-ml water 60-ml ethylene glycol 20-ml acetic acid 1-ml HNO_3	Acetic glycol etchant for pure Mg and alloys. Swab specimen 1–3 s for F and T6 temper alloys, 10 s for T4 and 0 temper alloys. Reveals grain boundaries in solution-treated castings and most wrought alloys.
10-ml water 100-ml ethanol 5-g picric acid	For Mg and alloys. Use fresh. Immerse specimen for 15–30 s. Produces grain contrast.

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[4] Buehler SUM-MET: *The Science Behind Materials Preparations*, Lake Bluff, IL, 2011; p 36 and p 73.

Table A-6. Leco metallography. Commonly used etchants for metals and alloys. Mg and alloys.[5]

Etchant: Composition	Metal	Procedure
1d: 200-ml water 1-ml HF	Mg-Zn-Zr Mg-Th-Zr	Swab with cotton for 15 s.
74a: 100-ml ethanol (95%) or methanol (95%) 1–5-ml HNO_3	Pure Mg Mg-Mn Mg-Al, Mg-Al-Zn (Al+Zn<5%) Mg-Al, Mg-Al-Zn (Al+Zn>5%) Mg-Zn-Zr Mg-Th-Zr	Etching rate is increased, selectivity decreased with increased percentage of HNO_3.
118: 25-ml water 75-ml diethylene glycol 1-ml HNO_3	Pure Mg Mg-Al, Mg-Al-Zn (Al+Zn<5%) Mg-Al, Mg-Al-Zn (Al+Zn>5%) Mg-Zn-Zr Mg-Th-Zr	Swab 3 to 5 s for F and T6; 10 s for T4 and 0 temper.
119: 20-ml water 60-ml diethylene glycol 20-ml acetic acid 1-ml HNO_3	Pure Mg Mg-Mn Mg-Al, Mg-Al-Zn (Al+Zn<5%) Mg-Al, Mg-Al-Zn (Al+Zn>5%) Mg-Zn-Zr Mg-Th-Zr	Swab 1 to 3 s for F and T6; 10 s for T4 and 0 temper.
120: 90-ml water 10-ml HF	Pure Mg Mg-Al, Mg-Al-Zn (Al+Zn>5%) Mg-Rare Earth-Zr Mg-Zn-Th-Zr	Immerse with gentle agitation 3–30 s.
121: 100-ml methanol (95%) or ethanol (95%) 4-g picric acid 0.7-ml H_3PO_4	Pure Mg Mg-Al, Mg-Al-Zn (Al+Zn>5%) Mg-Rare Earth-Zr Mg-Zn-Zr Mg-Th-Zr Mg-Zn-Th-Zr	Composition critical: (a) Immerse with gentle agitation 10–30 s. (b) To increase staining immerse and withdraw with a meniscus layer. Tease etchant over surface until dark stain develops.
122: 100-ml water 2-g oxalic acid	Pure Mg Mg-Mn Mg-Al, Mg-Al-Zn (Al+Zn<5%) Mg-Al, Mg-Al-Zn (Al+Zn>5%) Mg-Zn-Zr Mg-Th-Zr	Swab

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[5] Johnson, C. A. Metallography Principles and Procedures; Leco Corporation: St. Joseph, MI, 1992, p 38, pp 46–47.

Table A-6. Leco metallography. Commonly used etchants for metals and alloys. Mg and alloys (continued).

Etchant: Composition	Metal	Procedure
123: 100-ml ethanol (95%) 60-ml H_3PO_4	Pure Mg Mg-Al, Mg-Al-Zn (Al+Zn<5%)	Electrolytic; use stainless steel cathode. Space electrodes 2 cm apart. Start at 3 V DC. After 30 s maintain at 1.5 V.
124: 10-ml water 100-ml methanol (95%) or ethanol (95%) 5-ml acetic acid 6-g picric acid	Mg-Mn Mg-Al, Mg-Al-Zn (Al+Zn<5%) Mg-Al, Mg-Al-Zn (Al+Zn>5%) Mg-Zn-Zr Mg-Th-Zr	Immerse with gentle agitation 10–60 s.
125: 100-ml methanol (95%) or ethanol (95%) 10-ml acetic acid 6-g picric acid	Mg-Al, Mg-Al-Zn (Al+Zn<5%) Mg-Al, Mg-Al-Zn (Al+Zn>5%)	Immerse with gentle agitation 15–30 s.
126: 15-ml water 100-ml methanol (95%) or ethanol (95%) 30-ml acetic acid 6-g picric acid	Mg-Al, Mg-Al-Zn (Al+Zn<5%) Mg-Al, Mg-Al-Zn (Al+Zn>5%)	Immerse with gentle agitation 1–30 s.
127: 20-ml water 50-ml methanol (95%) or ethanol (95%) 20-ml acetic acid 3-g picric acid	Mg-Al, Mg-Al-Zn (Al+Zn<5%) Mg-Al, Mg-Al-Zn (Al+Zn>5%) Mg-Zn-Zr Mg-Th-Zr	Immerse with gentle agitation 5–30 s.
128: 200-ml water 8-ml HF 5-ml HNO_3	Mg-Zn-Zr	Immerse with gentle agitation 5–15 s.

List of Symbols, Abbreviations, and Acronyms

BEI	backscattered electron image
EDS	energy dispersive x-ray spectroscopy
MENA	Magnesium Elektron North America
Mg	magnesium
SE[M]	secondary electron mixed detector mode
SEI	secondary electron image
SEM	scanning electron microscopy
YAG	Yttrium-aluminum-garnet

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