

# Development of Rolling Schedules for Equal Channel Angular Extrusion (ECAE)–Processed AZ31 Magnesium Alloy Sheet

by Laszlo J Kecskes, Vincent H Hammond, Michael Eichhorst, Norman Herzig, and Lothar Meyer

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# Development of Rolling Schedules for Equal Channel Angular Extrusion (ECAE)–Processed AZ31 Magnesium Alloy

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Rolling procedures for equal channel angular extrusion (ECAE)-processed AZ31 magnesium plates have been developed.						
Using a temperature of 300 °C and a 10% reduction per rolling pass, the initially 9-mm-thick plates were successfully reduced						
into 1.5-mm-th	nick sheets. Two s	ets of plates, each v	with a different te	xture type, w	ere evaluated. Microscopic examination of	
the rolled sheet	t material shows a	highly heterogene	ous substructure,	consisting of	2 types of precipitate colonies dispersed in a	
refined grain si	ize (~10 μm) mag	nesium alloy matrix	x. Furthermore, th	he as-rolled n	nicrostructure is banded, containing	
alternating layers of coarser and finer grains. These regions are separated by a network of shear bands. Mechanical testing						
results show a relatively high strength and high ductility. However, the ability to fabricate sheet material from the ECAE						
along the longitudinal and transverse directions, respectively, than does texture C.						
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# 1 Introduction

This follow-on report builds on and continues the work established in a previous US Army Research Laboratory (ARL) technical report, "Development of Rolling Schedules for AZ31 Magnesium Alloy Sheets", ARL-TR-7277, June 2015, on further applying the experimentally developed rolling procedures to a series of AZ-31B magnesium plates that have been subjected to equal channel angular extrusion (ECAE) processing. Specifically, in the first report we documented the microstructural and mechanical properties of as-received AZ-31B tooling plate material, obtained from Magnesium Electron North America, Madison, IL. Because the tooling plate is received in an as-rolled condition, it has a fairly strong basal texture, which in turn limits one's capability to rolling to thinner sections. However, we demonstrated that in certain orientations, rolling to thin sections, as thin as 1.5 mm, is feasible. The report details the rolling schedules, temperature and reduction per pass, and the resultant macro- and microstructures.

Continued interest in magnesium and its alloys is attributed to the fact that it is the lightest metallic material with the potential for widespread structural applications. Its density is about a quarter of steel, and about two-thirds of aluminum, and its castability is also very good. With the appropriate suite of properties, magnesium alloys may find great utility in niche applications like personnel protection body armor, where low density and the reduction of mass are the most important criteria. With a potential for lowering the mass of the body armor package, the burden for Soldiers can be reduced significantly. To date, protective body armor plates or inserts have relied on metal-ceramic composite assemblies to reduce back-face penetration, wherein often aluminum is used as the metal component. Depending on its thickness and configuration, it is envisioned that with the application of magnesium, the mass of the metallic ceramic-structure could be decreased significantly.

For now, due to their good castability, most commonly available magnesium alloy components were produced by melting and casting. But often, sheet or plate material is necessary for use in structural applications. However, the required forming operations from melt-cast billets into sheet products are not straightforward. Wrought magnesium sheets having thin thicknesses are very difficult to produce because of the inherently low ductility of magnesium and its alloys, but in practice, the ductility of magnesium can be increased significantly with increasing temperatures. Therefore, in general, magnesium sheets are produced at temperatures higher than 300 °C. Unfortunately, at this temperature range, dynamic recrystallization processes take place, leading to a partial loss of mechanical properties.

In the previous report, we investigated the processability of as-received AZ31-B tooling plate in its initial state. The plates were prehomogenized at about 440 °C for 1 h and rolled at temperatures of about 425 °C afterwards. Thin sheets with a thickness of about 1.5 mm could be produced with very good surface quality without obvious cracks and. Specifically, these investigations identified an optimized sequential rolling procedure that could produce sheets with a thickness of about 1.5 mm and, more importantly, at lower temperatures of about 275 °C. However, small cracks on the edges were detected in the final sheets. Nevertheless, given the relative success of this initial rolling procedure, it was felt that it could be used as a baseline or reference in the current investigations to develop an equally viable rolling methodology for AZ31 plates that have been subjected to ECAE processing. The purpose of the ECAE process step is to reduce the texture anisotropy and refine the grain size of the as-rolled tooling plate material.

As such, the aim of the investigations described herein is to investigate the rollability of the magnesium alloy AZ31 to thicknesses of about 1.5 mm with the optimized rolling procedure and rolling temperatures of about 275 °C without destroying the ultra-fine-grained microstructure by suppressing or preventing grain growth. Concurrent with developing the procedure, evaluation of the microstructural and mechanical properties was of interest.

Eight ECAE-processed magnesium plates (AZ31) with 2 different textures (A and C) and a thickness of about 12.5 mm were delivered from ARL to Nordmetall GmbH, Adorf-Neukirchen, Germany, for the rolling experiments (Table 1). The length of the plates varied between 120 and 135 mm. Photographs of the asreceived plates are shown in Figs. 1 and 2.

Туре	Description	Thickness (mm)	Width (mm)
	Plate 10	12.5	153
Toutum A	Plate 15	12.5	153
Texture A	Plate 26	12.5	153
	Plate 32	12.5	153
	Plate13	12.5	153
Touture C	Plate 21	12.5	153
Texture C	Plate 25	12.5	153
	Plate 37	12.5	153

Table 1 Summary overview of the as-received plates



Fig. 1 Photographs of the as-received plates marked with "texture A"



Fig. 2 Photographs of the as-received plates marked with "texture C"

## 2. Experimental Setup

### 2.1 ECAE Procedures

Prior to ECAE extrusions, plates with nominally  $6 - \times 6 - \times 1/2$ -inch dimensions were sectioned from a much larger, as-received tooling plate. Figure 3 shows that the orientation of the ECAE plate normal was perpendicular to the rolling direction; that is, the ECAE plate normal was parallel to the hexagonal close packed c-axis. This orientation assured that the plates could be extruded without a potential for shear failure.



Fig. 3 Relationship between the tooling plate and ECAE plate orientations

ECAE processing entails the extrusion of metallic materials through a die with 2 intersecting channels, most frequently, at 90°. The input and output channels have the same dimensions; therefore, there is little postextrusion machining needed between consecutive passes. Two textures were created using a hybrid extrusion procedure. For the texture A plates, the plates were subjected to a sequence of 2 route A passes at 225 °C, one route C pass at 200 °C, one route A passes at 200 °C, and 2 route A passes at 150 °C. Conversely, for the texture C plates, the plates were subjected to a sequence of 2 route A passes at 150 °C. For route A, the plate is inserted without a change of its orientation on subsequent passes. For route C, the plate is rotated by 180° about the plate normal on the second pass. On the first 2 passes, the temperature

reflects a minimum value only, primarily, to prevent premature failure of the plate. That is, its value did not affect the subsequent processing steps if it was somewhat higher.

As the material passes through the ECAE tool, it first upsets, then shears roughly at  $45^{\circ}$  at the channel intersection. As such, during each pass each grain within the plate undergoes shearing. With each additional pass, the shearing texture is exaggerated and, unless reversed, an acicular texture in the material could result. The function of route C is to return the sheared grains back to their initial equiaxed state. However, it may be noted that during the shearing process, dislocations and dislocation networks are introduced, and the initial grains are refined.

Briefly, the ECAE procedures entailed preheating the plates in an external oven, then inserting it into the tool. The tool was preheated to the extrusion temperature. Thus, the plate could be further temperature equilibrated in the tool before extrusion was initiated. To reduce friction effects between the tool and the plate, initially, plates were lubricated with Loctite Brand Silver Grade Anti Seize compound, up to 700 °C (1,300 °F) (WW Grainger, Baltimore, MD) and wrapped in a 0.1-mm (0.004-inch)-thick sheet of Teflon-PTFE sheet (McMaster-Carr, Robbinsville, NJ). Subsequently, a better lubricant with higher temperature stability up to 1,200 °C (2,200 °F) was found (Never Seez, Anti-Seize and Lubricating Compound, High Temperature, Stainless, Nuclear Grade [Bostik, Inc., Middleton, MA]). Additionally, during the extrusions, backpressure was applied to confine and hold the plates in place. Otherwise, due to the limited number of slip systems or lack of availability of other, most likely, twinning-based deformation mechanisms activated in the magnesium alloy, premature shear of the plate would have occurred. The extrusion rate was set at or below 0.01 cm/s (0.004 inch/s) to ensure the plates did not shear prematurely. After extraction from the tool, the plates were allowed to cool. Usually, most of the Teflon layer was easily removed; however, on occasion small pieces of it were trapped whereby a series of surface fissures and folds along the edges and top and bottom surfaces of the plate were created. To prevent premature failure of the plates, these were removed, and the plate surface's was machined by milling before reinsertion and reextrusion.

During extrusion, the extrusion load as a function of the plate displacement was recorded. These data are very useful, as they illustrate the relative ease or difficulty of the extrusion or if there is any premature shear failure in the plate. These curves, are listed for the 8 plates in the Appendix. A cursory look of the graphs in Figs. A-1 through A-8 shows that none of the plates experienced any major deviations from what is considered to be typical of magnesium at elevated temperatures. Ideally, the load is expected to be uniform as the plate transits through the input and output channels. Any sudden drop in the load corresponds to shear failure of the plate by

shear banding. Conversely, an increase in the load, gradual or sudden, implies nonuniform extrusion conditions, wherein, a part of the plate is, most likely, experiencing greater friction loads than normal. (This could be simply an artifact of the Teflon wrapping slipping off or insufficient lubrication of the internal surfaces.) Nominally, for most of the plate extrusions a steady state condition existed with mostly flat loads that varied between 355 and 800 kN (80 and 180 klbf) with the average being about 535–575 kN (120–130 klbf). However, in some cases, when friction effects were not reduced, the peak load was as high as 890–980 kN (200–220 klbf). Usually this occurred as the tail end of the plate was extruded before the entire plate was in the exit channel.

#### 2.2 Hot Rolling

The rolling experiments were performed in cooperation with the Technical University Bergakademie Freiberg, a leading university in Germany for the production of magnesium sheet materials. After preheating, the initially 12.5-mm-thick plates were rolled to a thickness of about 1.5 mm. The hot rolling was performed on a dual-rolling mill (Hugo Sack Mills, formerly of Dusseldorf, Germany) (Fig. 4). The diameter of the roller was about 360 mm, with a maximum rolling force of 2,400 kN. The rollers are driven by a 160-kW electric motor capable of producing a torque of 30 kNm. The rollers were preheated to about 120 °C with electric blankets before the rolling experiments, and a lubricant was sprayed on the rollers before each rolling pass. The rolling speed was about 1 m/s, a common rolling speed used for AZ31. The variations in rolling passes are described later in the respective experimental sections.



Fig. 4Photograph of the rolling mill (Hugo Sack GmbH)

The preheating (15–20 min) and annealing between the rolling steps was performed in a convection air furnace (Fig. 5). Unlike that in the previous experiments, for the ECAE plates, a homogenization annealing was not performed before starting the rolling experiments.



Fig. 5 Convection air furnace

Starting from our prior results, the first rolling passes started with a temperature of about 275 °C. However, due to cracks on the edges and surface cracks, the temperature was increased to about 300 °C for further rolling passes.

#### 2.3 Metallographic Investigation

Representative specimens were taken from the transverse and longitudinal directions of the as-received plates (Fig. 6). One plate of each texture (texture A, plate 32; texture C, plate 37) was used for preparation of microsections. The results of the rolling experiments were investigated by examining the longitudinal microsections taken from the upper and middle zones of the sheets (Fig. 7). The specimens were mounted in an epoxy resin and prepared using conventional metallographic techniques. A final etching, using diluted picric acid, was used to better reveal the underlying grain structure. The grain size was characterized and measured by the linear interception method applied across the grain boundaries.



Fig. 6 Illustration of the microsection positions in the as-received plates



Fig. 7 Illustration of the microsection positions of the rolled sheets

#### 2.4 Quasi-Static Tensile Tests

The tensile tests at quasi-static strain rates  $(10^{-3} \text{ s}^{-1})$  and room temperature were performed using a universal testing machine built by the Zwick/Roell company. The maximum force of the testing machine is 100 kN, and the testing velocity can be varied between 0 and 500 mm/min. The experimental setup is shown schematically in Fig. 8. The testing machine consists of a stiff frame, 2 ball bearing screw spindles, and a crosshead connected to the ball bearing screw spindles. The forces were measured with a load cell with a maximum force of 5 kN. The elongation was measured with a fine displacement extensioneter.



Fig. 8 Universal testing machine (schematic)

One plate in the as-received condition of each texture (texture A: plate 15, texture C: plate 13) was used to fabricate tensile specimens in both longitudinal and transverse directions. The tensile specimen geometry is shown in Fig. 9. The gage length is about 10.5 mm.



Fig. 9 Tensile specimen (as-received material)

After rolling to about 1.5 mm, tensile specimens with a gage length of about 15 mm and a width of about 5 mm were manufactured (Fig. 10), and tensile tests were performed using the Zwick/Roell universal testing machine.



Fig. 10 Tensile specimen (as-rolled material)

## 3. Results and Discussion

#### 3.1 Investigation of the As-Received Materials

The surface quality of all as-received plates was quite poor. Cracks with depths up to 2–3 mm were found on nearly each plate. As indicated in the experimental section, the origin of the cracks are likely attributed to the use of a Teflon sheet to reduce friction, which, in turn, was incorporated into the shear zones. Common surface defects are illustrated in Fig. 11 for 2 representative plates from each texture type.



Fig. 11 Photograph illustrating the surface quality of the as-received plates

The results of the tensile tests from the texture A and texture C specimens are illustrated in the engineering stress-engineering strain diagrams in Figs. 12 and 13.



Fig. 12 Engineering stress vs. engineering strain diagram for the texture A, plate 15 tensile specimens.



Fig. 13 Engineering stress vs. engineering strain diagram for the texture C, plate 13 tensile specimens.

Comparing the stress-strain curves for texture A and texture C there is a higher ductility in the longitudinal and transverse directions of the texture C specimen. Furthermore, the ductilities of the texture C specimen for the longitudinal and transverse directions, respectively, are closer to one another. This is not the case for the texture A specimen, which is considerably less ductile in the longitudinal direction. On the other hand, however, the strength difference of the texture A specimens, between the longitudinal and transverse directions, is less than that for the texture C specimens.

Microsections of the longitudinal and transverse directions of texture A, Plate 32, (Fig. 14) and texture C, Plate 37, (Fig. 15) were prepared.



Fig. 14 Microsections of the as-received texture A, plate 32



Fig. 15 Microsections of the as-received texture C, plate 37

Figs. 14 and 15 illustrate that the majority of the magnesium grains are very small and could not be resolved and thus could not be investigated in detail by optical microscopy. Nevertheless, it is apparent that both texture types exhibit a highly banded or fibrous substructures of alternating layers of coarser and finer grains. A comparison of the 2 types of textures reveals that texture A is less uniform than texture C. However, this is as expected, as banding ought to be more pronounced in the texture A type plates. Further comparisons of the inner and outer zones of each type reveals that, generally, their microstructures are very similar across the plate width.

Aside the overall features of the plate textures, the microstructure contains large colonies of 2 types of precipitates: those that etch black and those that etch white. The larger white grains have diameters of about 50  $\mu$ m or larger; a cluster is illustrated in Fig. 16. Additionally, due to the high degree of deformation imparted by the ECAE processing, many shear bands were observed in the plate sections (Fig. 17). It is believed that the shear bands contain very fine micrometer-sized recrystallized grains. Lastly, in all of the microsections a second series of black precipitates were also observed (Fig. 18). These precipitates are finer, only 10–20  $\mu$ m, and lie along the shear planes, but are a little more uniformly dispersed than their white counterparts. In particular, a more detailed investigation should be done on their chemical composition in further studies.



Fig. 16 Large white grain clusters in a microsection of the texture A, plate 32; longitudinal section



Fig. 17 Shear bands visible in a microsection of the texture C, plate 37; longitudinal section



Fig. 18 Dark precipitates in a microsection of the texture C, plate 37; transverse section

## 3.2 Hot Rolling of the Plates

Because of the poor surface quality of the plates, about 3 mm from the top and bottom surface were removed by milling. Additionally, the front and back end of each plate (about 15 mm) was also cut off. Thus, the rolling experiments were initiated with a plate thickness of about 9 mm and a plate length of about 100 mm.

Photographs of the plates with reduced dimensions are shown in Figs. 19 and 20; the rolling direction is marked in black ink and indicated by the arrow. Despite the removal of the exterior layer, some of the cracks on the as-machined surface could be still observed.



Fig. 19 Photographs of the texture A plates, prior to the rolling experiments



Fig. 20 Photographs of the texture C plates, prior to the rolling experiments

The rolling experiments started with a preheating of the plates at about 275 °C for about 15–20 min in the convection air furnace, followed by 9 rolling passes and 5 intermediate annealing steps. The data for plate A\_32 and plate C\_37 are shown in Table 2. The degree of deformation after every rolling pass was about 15%. Initially, after 9 rolling passes to a thickness of about 1.6 mm, cracks on the edges of the resultant sheets could be observed for both texture types (Fig. 21, plate A\_32 and plate C\_37). Therefore, the degree of deformation was decreased to about 10% per rolling pass. The cracks on the edges decreased significantly for both textures (Fig. 22, plate A\_26 and plate C\_25). The sheet resultant from plate C\_25 is nearly free of cracks and shows, compared with all of the other rolled sheets, the best surface quality. To further decrease the cracks on edges for texture A, the rolling temperature was increased to about 300 °C and a degree of deformation per pass of about 10%.

The results are also illustrated in Fig. 21. Smaller cracks on the edges can be still observed for texture A compared with the other rolling parameters. For Plate C\_21 the cracks on the edges are larger compared with those for the sheet obtained from plate C\_25. It is hypothesized that this is mainly caused by a worse initial quality of plate C\_21.

	Plate Number					
<b>Rolling Pass</b>	A_32	A_26	A_10	C_37	C_25	C_21
1	8.4	7.9	8.	7.5	8.1	8.1
2	7.2	7.5	7.4	6.5	7.2	7.3
3	5.95	6.9	7.0	5.7	6.8	6.8
4	4.95	5.9	6.1	4.7	5.8	5.9
5	4.00	5.3	5.4	4.1	5.4	5.4
6	3.60	5.0	5.0	3.4	4.9	5.0
7	3.00	4.5	4.6	2.9	4.4	4.5
8	2.40	4.0	4.2	2.3	4.0	4.1
9	1.60	3.5	3.6	1.6	3.6	3.6
10		3.2	3.3		3.2	3.2
11		2.7	2.9		2.9	2.9
12		2.3	2.3		2.4	2.5
13		1.8	1.9		1.9	1.9
14		1.5	1.5		1.6	1.5

 Table 2
 Rolling conditions for the ECAE processed plates

Furnace Temperature:	275°C	275°C	300°C
Degree of Deformation			
per Rolling Pass:	≈15%	≈10%	≈10%

- - - - intermediate annealing at 275/ 300°C for 15-20 minutes



Fig. 21 Macro-photographs of the rolled sheets

If the rolling results of texture A and texture C are compared with each other, the quality of the sheets with texture A appears to be worse. An explanation might be that the poorer and more nonhomogenous ductility of the initial texture of the texture A plates is not optimal for further rolling processes (Fig. 12). The poorer ductility, especially, that in the longitudinal direction, could be a result stemming from the orientation of the magnesium basal plane after ECAE processing as determined by the cumulative effect of multipasses of route A.

The microstructure after rolling to a thickness of about 1.5 mm was investigated in the middle and upper zone of the sheets. All microsections were cut from the longitudinal direction. A representative microstructure for texture A is shown in Figs. 22 and for texture C is shown in Fig. 23.



Fig. 22 Microstructures in the longitudinal direction of the 1.5-mm sheets of texture A

After rolling, areas with greater degree of deformation can be observed for all examined sheets. The as-rolled microstructure is characterized by recrystallization processes that resulted in very fine grain sizes. Between the zones with a high

degree of deformation, larger grains with about a  $10-\mu m$  grain size can be found. Within these regions of larger grains, small twins could be observed; these were formed due to the low rolling temperature and high degree of deformation.



Fig. 23 Microstructure in the longitudinal direction of the 1.5-mm sheets of texture C

Tensile specimens in both transverse and longitudinal directions from one of the rolled sheets (texture A, sheet 26; texture C, sheet 25) representative of each texture were fabricated. The results of the quasi-static tensile tests of the as-rolled material are illustrated in Figs. 24 and 25, respectively. Because of the different specimen geometries, the results from the as-received material and the as-rolled material are not comparable. However, semi-quantitatively, it may be concluded that after

rolling both texture types exhibit nearly the same strength (ultimate tensile strength [UTS] ~280–300 MPa). The decrease in UTS compared to the as-received material is due to the observed recrystallization and associated loss of defects, introduced by the deformation processing. There is a decreased elongation to failure in the longitudinal direction for the texture A (~12%) results. In contrast, texture C is characterized by a higher elongation to failure of about 17%–20% in both transverse and longitudinal directions.



Fig. 24 Engineering stress vs. engineering strain diagram for texture A; as-rolled material (plate 26)



Fig. 25 Engineering stress vs. engineering strain diagram for texture C; as-rolled material (plate 25)

#### 4. Summary

Rolling experiments were conducted on AZ31B magnesium plates, with 2 types of textures, which were subjected to ECAE, using a novel hybrid route methodology. The rolling experiments with a rolling temperature of about 275 °C, a degree of deformation of about 10% per rolling pass, and a rolling speed of about 1 m/s showed very promising results. The best results were obtained for plates with texture C, especially, for plate 25. After final rolling, this sheet with a thickness of about 1.5 mm was nearly free of cracks and could be characterized by uniform tensile properties in the transverse and longitudinal directions and having a recrystallized but very fine microstructure. The good quality of the rolling results for this plate can be explained in terms of it having the best surface quality of the initial as ECAE-processed plate and the good isotropic ductility in the transverse, especially in the longitudinal directions. In contrast, the results for texture A were not as good quality, which was attributed to the strong orientation asymmetry of its tensile properties, especially, in the longitudinal direction.

# 5. Conclusions and Prognosis

Eight ECAE-processed plates were sent from ARL to Nordmetall GmbH for rolling experiments. Four plates were marked with texture A and 4 plates were marked with texture C. The as-received material was characterized by macro- and microphotographic documentation and quasi-static tensile tests. Generally, a large number of cracks and bad surface quality of the as-received material were found during the initial evaluation. Specimens were prepared from one plate for each texture and tensile tests were performed in the longitudinal and transverse directions. The mechanical testing results pointed out that texture C was characterized by slightly higher strength and more isotropic or homogenous ductility in the longitudinal and transverse directions.

To reduce the influence of surface cracks, before starting the rolling experiments about 2–3 mm of each plate's surface was removed by milling. The rolling experiments were then performed using 3 different rolling parameters (Table 3). After rolling using the first variant, sheets with a thickness of about 1.6 mm, which had cracks on the edges, were fabricated. By decreasing the degree of deformation per rolling step and increasing the rolling temperature, the cracks on the edges could be reduced significantly. It could be also observed that the surface quality after rolling for texture C is much better than the surface quality of texture A. However, the most influencing parameter was determined to be the quality of the initial as-received material. Therefore, the best surface quality after rolling was observed to be for the second rolling variant for texture C.

Variant	Rolling speed (m/s)	Rolling temperature (C°)	Degree of deformation per rolling step (%)
1	1	275	15
2	1	275	10
3	1	300	10

Table 3Overview of the rolling parameters

As such, the best rolling results could be achieved by the following conditions:

- High surface quality of the initial material before rolling.
- A good and isotropic or homogenous ductility in both the longitudinal and transverse directions (similar to qualities exhibited by texture C).
- Using the rolling parameters according to the second variant (1 m/s, 275 °C, 10%).

It has to be emphasized that the current investigation was mostly focusing on the ability to roll the plates and the correlation of this capability with the microstructures and mechanical properties of the magnesium alloy material in the as-received and as-rolled condition. However, the most important insight into the feasibility of the rolled sheets in an end application are its mechanical properties. Therefore, further mechanical tests under different loading conditions (tension and compression) and at quasi-static and dynamic strain rates for the material in the as-received and as-rolled condition should be performed.

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Appendix. Load vs. Displacement Curves for the Texture A and Texture C ECAE Plates



Fig. A-1 Plate 10-texture type A extrusions: nos. 1–6



Fig. A-2 Plate 15-texture type A extrusions: nos. 1–6





Fig. A-4 Plate 32-texture type A extrusions: nos. 1–6



Fig. A-5 Plate 13-texture type C extrusions: nos. 1–6



Fig. A-6 Plate 21-texture type C extrusions: nos. 1–6





Fig. A-8 Plate 37-texture type C extrusions: nos. 1–6

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