



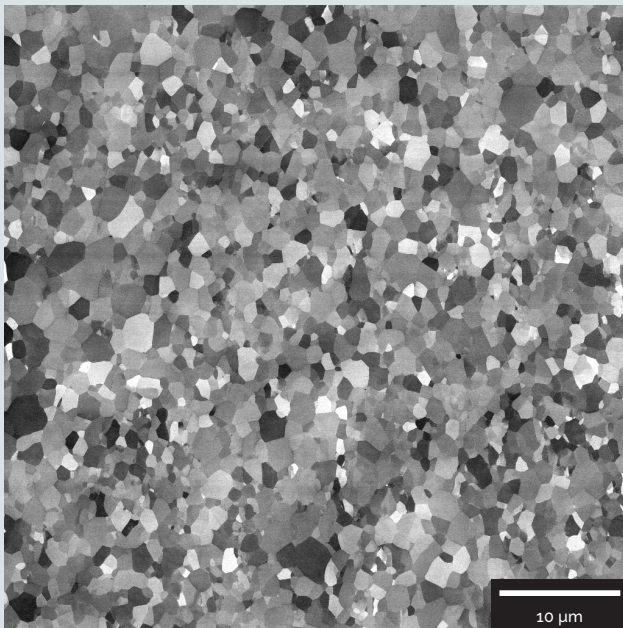
SEM and EBSD analysis of the grain structure after ECAP process of the aluminum material

The excellent casting properties of aluminum in combination with easy availability makes aluminum one of the most widely used metals in the world. The combination of light weight and excellent mechanical properties makes aluminum an ideal material for automotive and aeronautical applications as well as more basic uses. Aluminum has very good shaping properties, for example for rolling manufacturing. It is widely used in the food industry as a packaging material and in the building industry for the manufacturing of windows and doors. To enhance its material properties, aluminum can be tempered or alloyed by the addition of copper, zinc, magnesium, manganese, or silicon. Another approach is to modify the microstructure by applying one of the processes that induces internal strain such as extrusion, rolling, ECAP, and others.

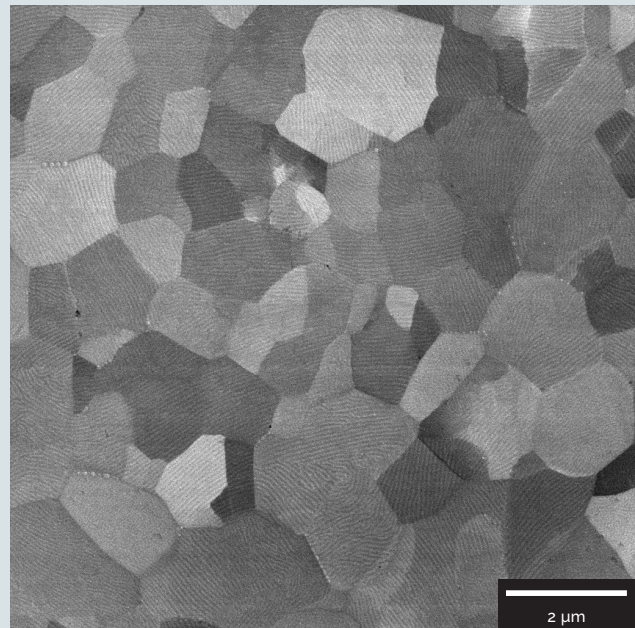
Equal channel angular pressing (ECAP) is a method using the principle of severe plastic deformation. The bulk material is extruded to create an ultra-fine grain structure. ECAP

is a metal forming process which refines grain structure to submicron or to nano grain scale and implements the high strain and superplasticity to the material. Material processed using the ECAP method shows an improvement of the mechanical properties.

ECAP is usually done in multiple steps and it is crucial to track the grain size and the morphology after a certain number of ECAP steps. Electron Backscatter Diffraction (EBSD) gives crystallographic information about sample's microstructure. This method uses the specific signal in scanning electron microscopy (SEM) which is detected by dedicated EBSD detector. This technique not only provides grain size information; the detected diffraction pattern also enables determination of the phase, crystal orientation, grain morphology and boundaries. Therefore, this SEM based technique is ideal and widely used in many applications and fields from standard metal research and geology to the industrial research in fields of nuclear, automotive, aerospace research and many others.



c **Fig. 1:** Channeling contrast of the sample treated by 8 steps of ECAP.

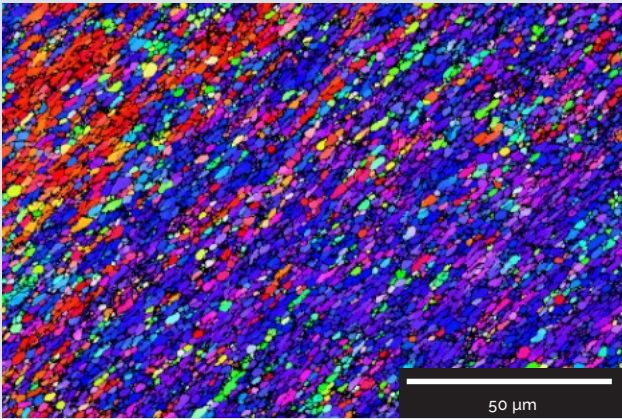


c **Fig. 2:** Channeling contrast combined with the sample surface topography information of the sample treated by 8 steps of ECAP.

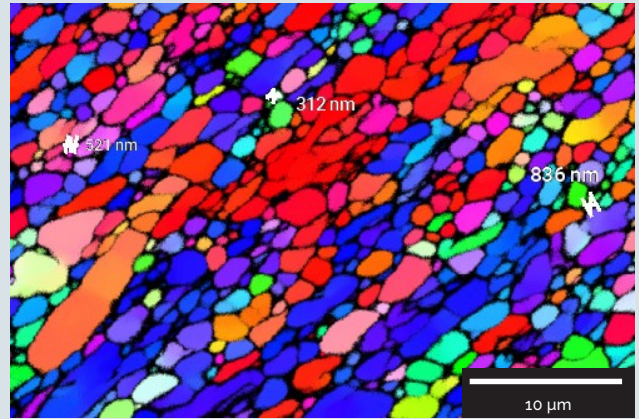


In our study the Field Emission Gun (FEG) Ultra High-Resolution Scanning Electron Microscope (UHR-SEM) TESCAN CLARA was used to determine the fine grain structure after the ECAP process. The TESCAN CLARA is a UHR-SEM using BrightBeam technology, allowing field free sample characterization and observation of the sample. Figure 1 illustrates the use of channeling contrast to reveal the different crystal (lattice) orientations of individual grains. Such large-area grain-orientation images are very helpful in localizing areas of interest for further crystallographic analysis. The channeling contrast mixed with the SE topography information can be seen in the figure 2. TESCAN's in-chamber ET (Everhart-Thornley) detector collects both secondary and wide-angle backscattered electrons, providing excellent topographic contrast. Therefore the channeling with fine details of the material structure are visible in figure 2. The optimal analytical and imaging conditions are set by In-Flight Beam Tracing™. The EBSD analysis of the Al sample was done at 20 kV acceleration voltage and 3nA beam current. TESCAN's real-time Spot Optimization function ensures that the best beam profile and optimal spot size are obtained even at high beam currents. This function is beneficial mainly

for analytical purposes where high beam currents are essential to providing sufficient signal for fast, statistically valid analyses. The spot size optimization is beneficial for analyzing larger areas at maximum speed and for precise high-resolution mapping as well. Our application example demonstrates the analysis of an Al structure after 8 steps of ECAP by EBSD mapping. EBSD was used to map two areas on the sample. The map, which covers a larger area (150 x 100 μm), is more suitable for defining the grain texture. Figure 3 shows an Inverse Pole Figure image colored in direction Y (IPF Y). This image shows the small grains which were created by ECAP and their crystallographic orientations. During the pressing of the material the preferential grain orientation can be created. To evaluate the grain orientation it is necessary to analyze a large number of grains to get statistically valid data. The grain orientation can be visualized for example by pole figures. The pole figures illustrate the crystal orientation by projection of the crystal orientation to the point. The grain structure is shown in figure 4. using the data from IPF Y map again. This precise mapping can reveal very fine grain structures with micron and even sub-micron grain sizes.



c Fig. 3: IPF Y map of the area 150 × 100 μm. Shows non-distorted, EBSD mapping at high spatial resolution.



c Fig. 4: IPF Y map of the small area 40 × 25 μm, Where grains of few hundreds of nanometers are shown analyzed at high current.



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