Fabricating Nanostructured Materials through Severe Plastic Deformation (SPD) Processing

Prof. Dr. Afërdita Priftaj Veveçka

Prof. Dr. Jorgaq Kacani

Polytechnic Universitety of Tirana,
Department of Physics

Albania


2007

Nanomaterials obtained via refining

- The strength of all polycrystalline materials is related to the grain size, *d*, through the Hall-Petch equation:

$$\sigma_y = \sigma_0 + k_y d^{-1/2}$$

d - grain size

- Definitions:
  - Bulk nanstructured materials (BNM) are defined as bulk materials having fully homogeneous and equiaxed micro structures with GS < 1μm and with grain boundaries having high angles of misorientation

  - *Ultrafine-grained materials (*UFG) (100 nm < *d* < 1μm)
  - *True nanocrystalline materials (d* < 100 nm)

Two main approaches to produce nanocrystalline materials

1. Bottom-up

- Inert gas condensation (Gleiter, 1984)
- Electrodeposition, (Erb et. al, 1989)
- Consolidation of nano-powders (Koch, 1990)
- Crystallization from amorphous materials

2. Top-down

Severe Plastic Deformation, "SPD" (Valiev et al, 1991, 1993) :

BULK nc materials

Severe Plastic Deformation (SPD)

- SPD - a metal forming procedure in which a very high strain is imposed on a bulk solid without change in the overall dimensions, leading to the production of exceptional grain refinement. This allows for the repeated application of the process to accumulate larger strains
- Due to their refined microstructure (small GS) they provide attractive properties that cannot be achieved in conventional GS materials with the same chemical composition.

Outstanding Properties of Bulk NanoMaterials (BNM)

These nanostructures lead to changes in physical and mechanical and other properties:

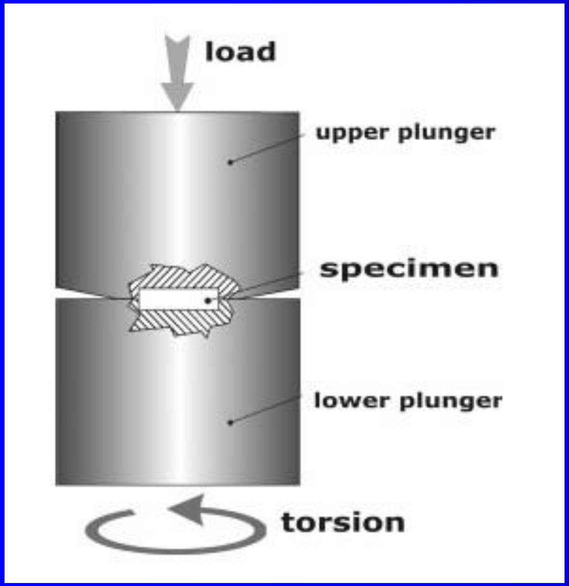
- high strength at low temperature and at good ductility
- high- speed and low –temperature superplasticity (SPD)
- improved magnetic properties
- Improved corrosion
- The microhardness of BNS materials is higher than that of CGS analogs by a factor of 2-7

Standard SPD Techniques

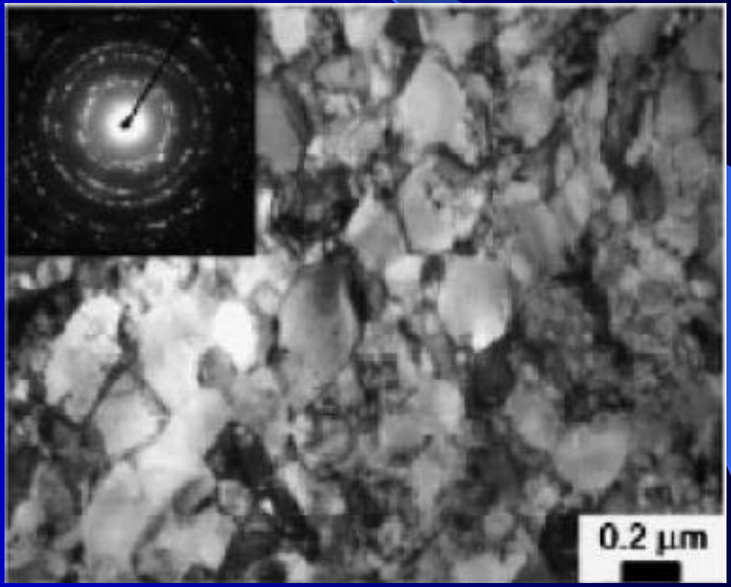
- Equal Channel Angular Pressing (ECAP)
- High Pressure Torsion (HPT)
- Accumulative roll-bonding (ARB)

High Pressure Torsion

Disc sample is torsionally deformed under high pressure of several GPa.



Schematic of HPT set-up (N.Bridgman, R.Valiev)



TEM microstructure of pure Ni produced by HPT, for N = 5 at applied pressure of 9 GPa.

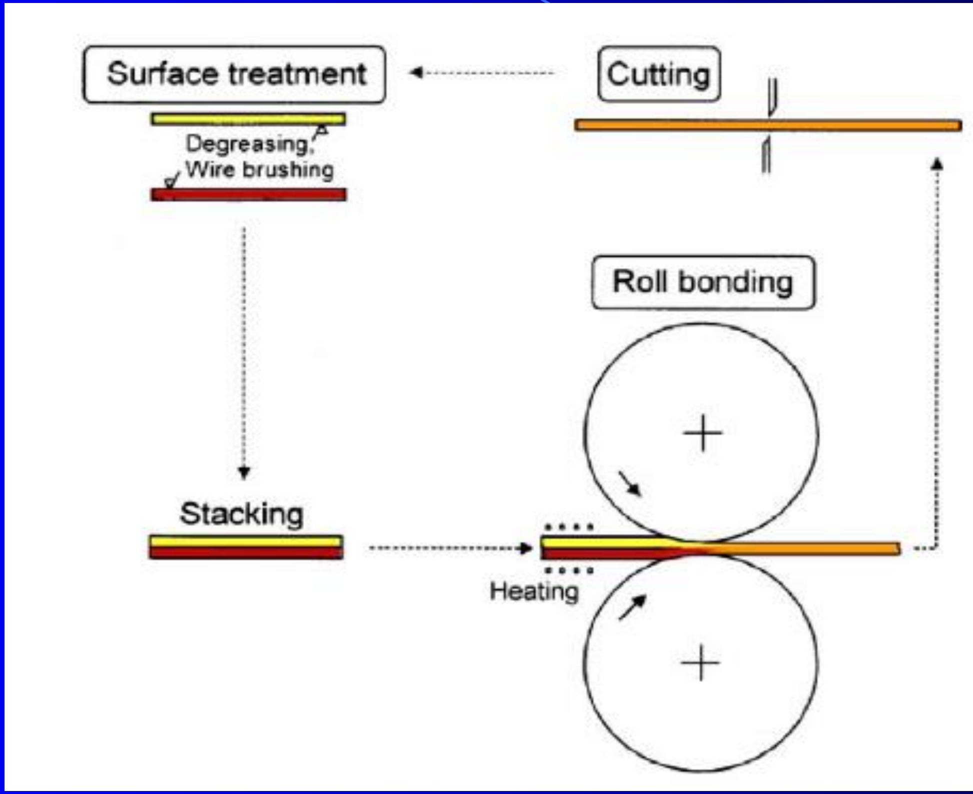
GS ~ 100 nm

Main properties of HPT technique

- Advantages:
  - gives high quality UFG materials (GZ<100 nm)
  - process brittle materials as intermetallics and semiconductors.
- Disadvantage :

  the specimen dimensions are fairly small, with max disc diameter ~ 20mm and thickness of ~ 1mm; limited industrial use

Accumulative Roll-Bonding (ARB)



Is the only SPD process using rolling deformation-invented in 1998 in Japan (N.Tsuji)

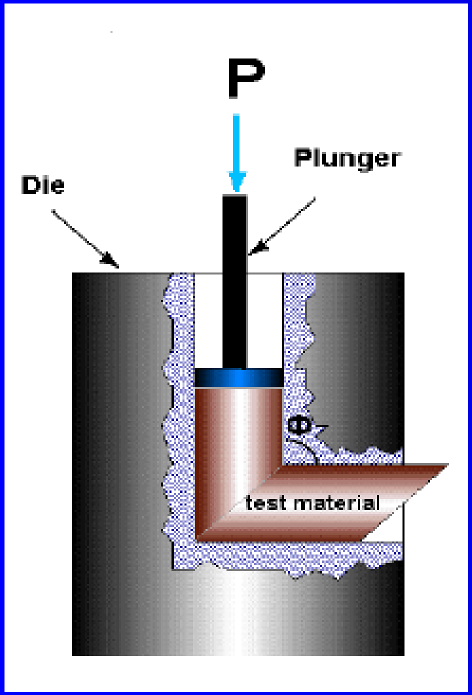
Main properties of ARB

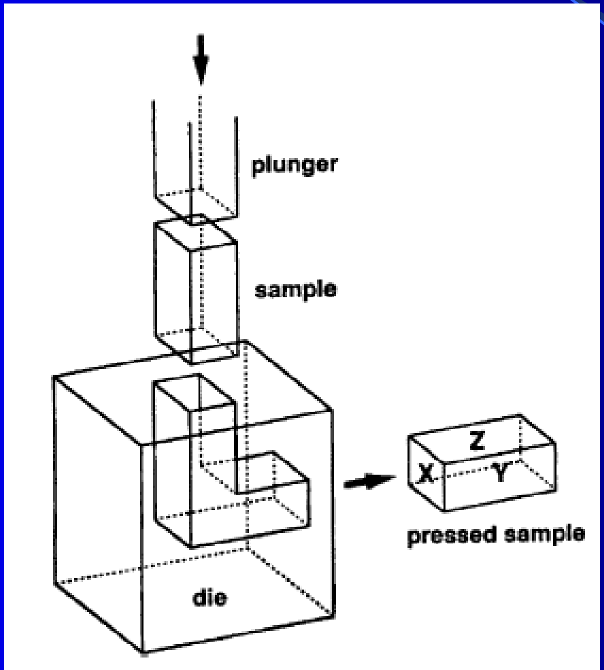
- To obtain one-body solid materials, the ARB is not only a deformation process, but also a bonding process (roll-bonding).
- To achieve good bonding, the surface of the materials is degreased and wirebrushed before stacking, and the roll-bonding is carried out at elevated temperatures.
- Depending upon the crystal structure, the microstructures have GS within the range of ~ 70-500 nm.
- The ARB materials have very high strength; 2 - 4 X higher than those of the same material with CGS

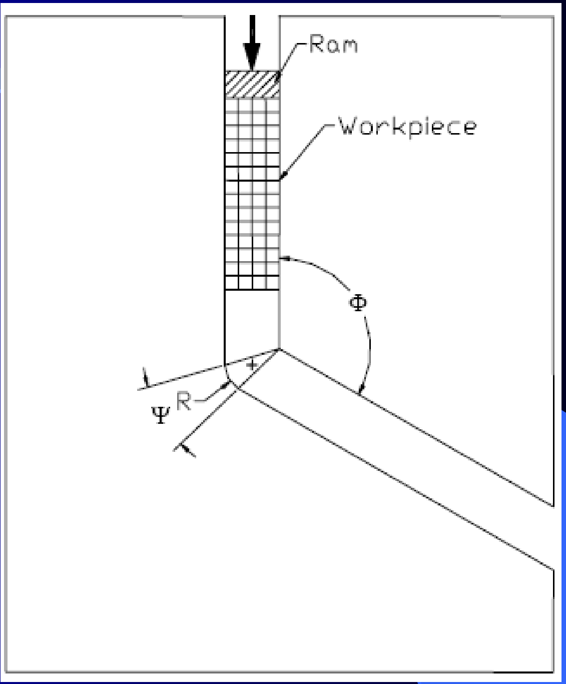
Equal Channel Angular Pressing (ECAP)

- First introduced by Segal and co-workers in 1980 in the former Soviet Union.
- After 1990 initiated an intense attention in the scientific community.
- Can be applied to fairly large billets
- Is a relatively simple procedure
- May be developed to materials with different crystal structure
- Reasonable homogeneity is attained

ECAP



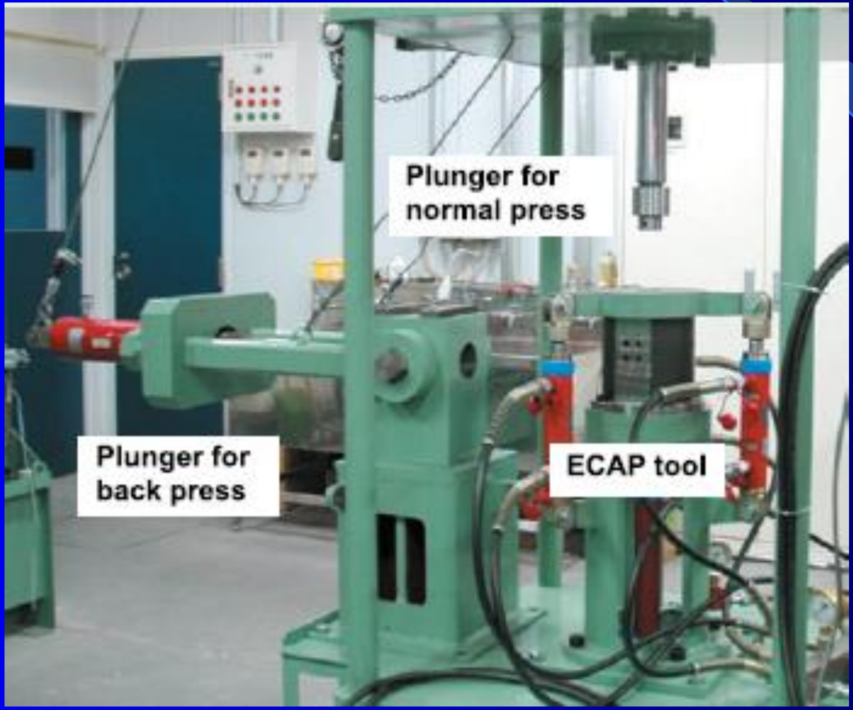


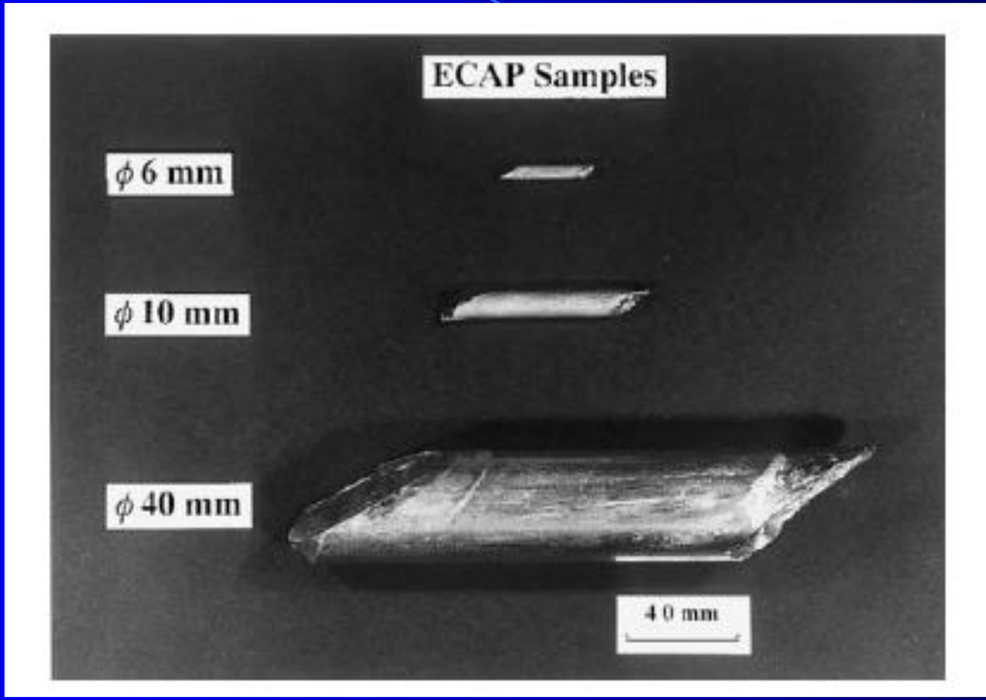


Schematic view of ECAP die

A section through the ECAP die: the internal angles ϕ, ψ.

View of ECAP facility





Typical appearance of the samples of Al – alloy after ECAP in dies with diameters 6, 10, and 40 mm.

Fundamental parameters in ECAP

Operates in simple shear and characterized by:

- Strain imposed in each separate passage through the die
- The slip systems operating during pressing

All these parameters play a critical role in determining the nature of UFG structure introduced by ECAP

The equivalent strain :

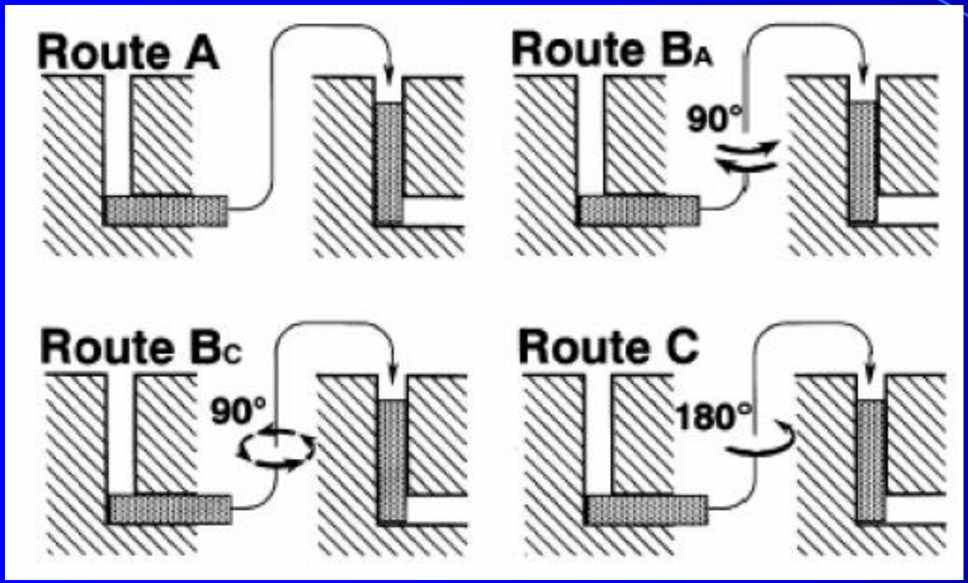
$$\varepsilon_N = \frac{N}{\sqrt{3}}\left[2\cot\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right) + \Psi \operatorname{cosec}\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)\right]$$

N is the number of passing through the die (assume no friction)

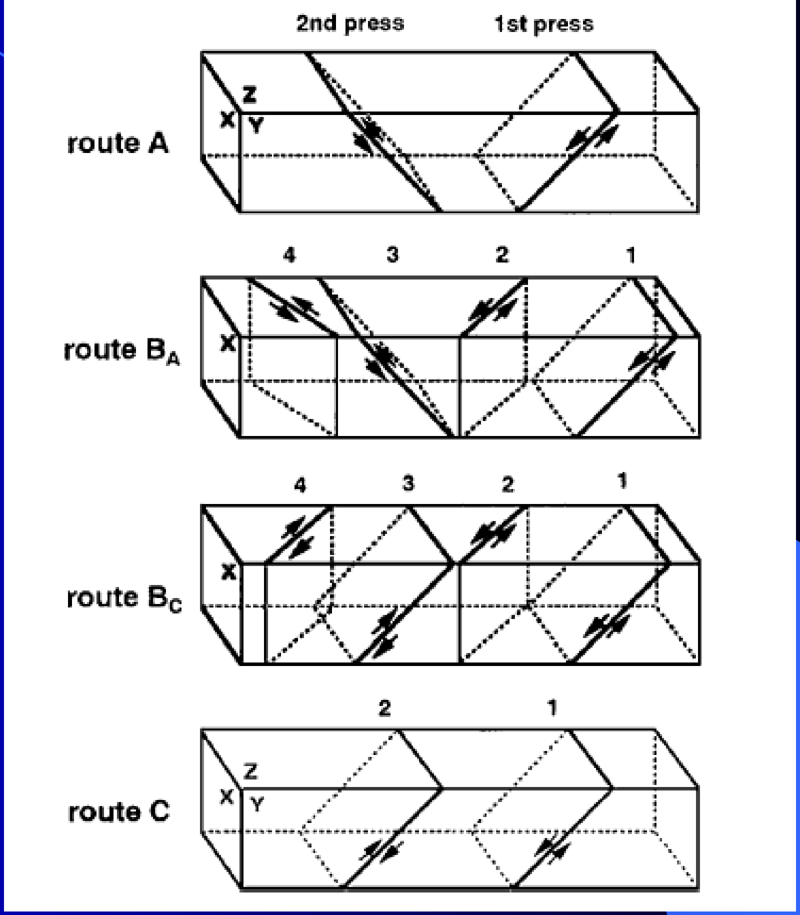
- For conventional dies, the angle $\phi = 90^o$
  $\varepsilon_N \sim 1$, for a single passage, for all values of ψ.

- Since the cross sectional shape of the work piece does not change, it can be introduced into the die and pressed again. Multiple passes results in a large accumulated strain.

The processing routes for ECAP



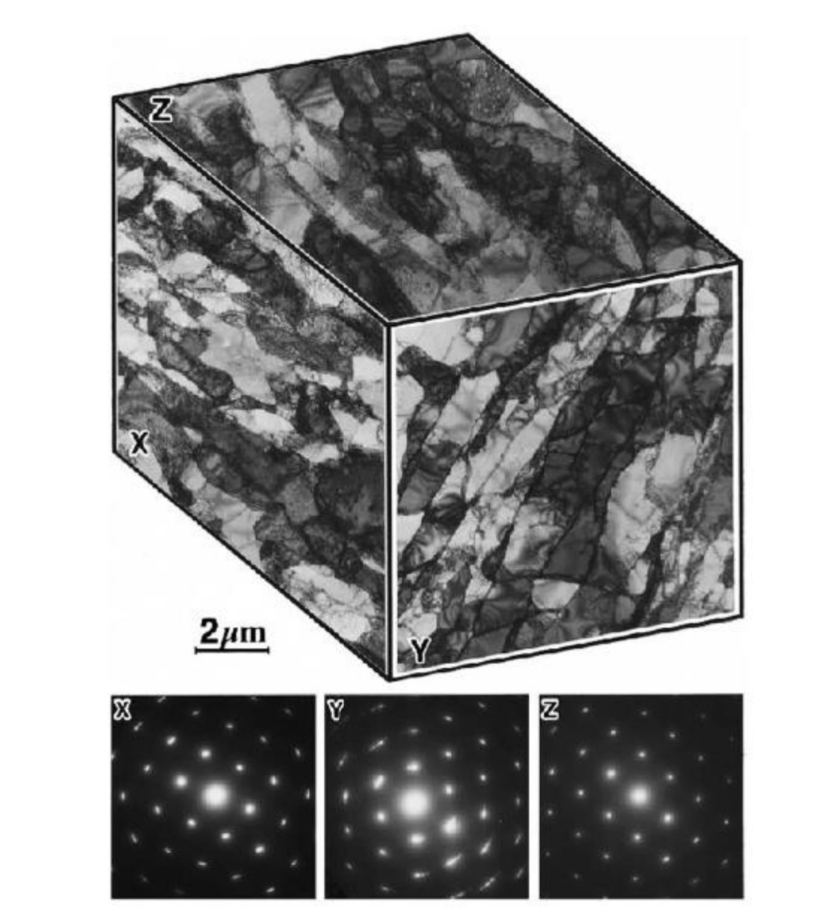
The best route for UFG
microstructure is route Bc



The slip systems viewed on the X, Y and Z planes for consecutive passes using processing routes

The development of UFG microstructure

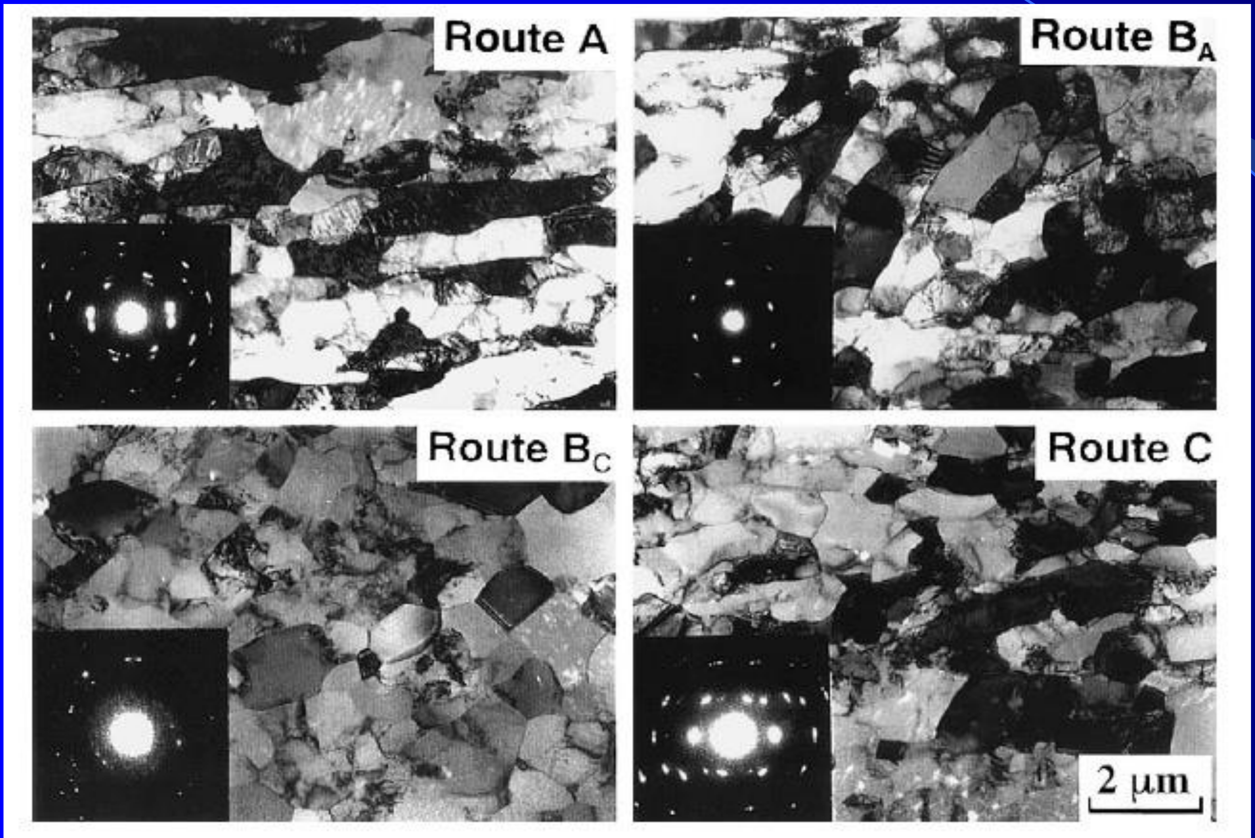
- Microstructures on the *X*, *Y*, and *Z* planes for polycrystalline AL after ECAP through 1 pass :



- Grains that are elongated
- Formation of low angle cell structure at the initial stages of SPD process in pure metals independent of their crystal lattice type.
- SAED patterns exhibit net patterns, indicative of low angle boundaries.

Y.Iwahashi, Z.Horita, M.Nemoto, T.G.langdon, Acta Mater. 1998,46

Appearance of the microstructures on the *X* plane



Polycrystalline Al, after ECAP through 4 passes

SAED: The spots are distributed around circles showing the presence of high angle grain boundaries

Route B_C leads to the most rapid evolution into an array of high angle boundaries

Microstructural features after ECAP

- An increase in ECA pressing leads to a gradual transformation of the low angle subgrain structure to the equiaxed grain nanostructure with high angle grain boundaries.
- This evolution is interpreted by rearrangement and annihilation of dislocations with opposite signs.
- The process of nanostructured formation during SPD is rather complex and its mechanisms is not yet fully explained

Some application of BNM

- UFG Ti and Ti alloys have entered the bio-medical market
  - Are currently used as implant materials in traumatology, orthopaedics and dentistry:
  - Due to their excellent biological compatibility, good corrosion resistance and high specific strength compared with other metals
- Nanostructured light alloys (Al and Mg alloys) for automotive industry

Improvement of mechanical properties of commercial Al alloy processed by ECAP

Objective:

- To examine the potential for using ECAP to refine the grain size and strengthen the commercial Al alloys

- The experiment was realized in collaboration with the department of Mechanics, University of Ancona, Italy

Experimental material and procedures

- **Al-5754** commercial alloy with composition in wt% :
  2.4-2.6% Mg, 0.1-0.6% Mn, 0.4% Cr, 0.4% Fe, 0.4% Si, 0.2% Zn with the balance Al.

- Alloy used for producing automotive parts and supplied by STAMPAL (Torino, Italy).

- Observations by optical microscopy revealed a grain size of 70 μm in the as received condition

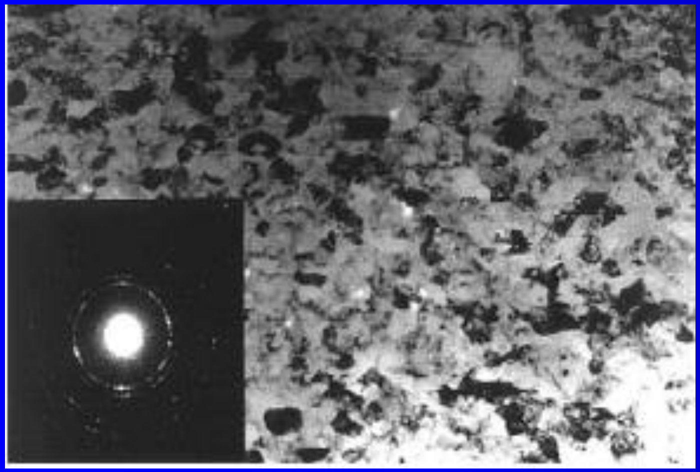
We studied

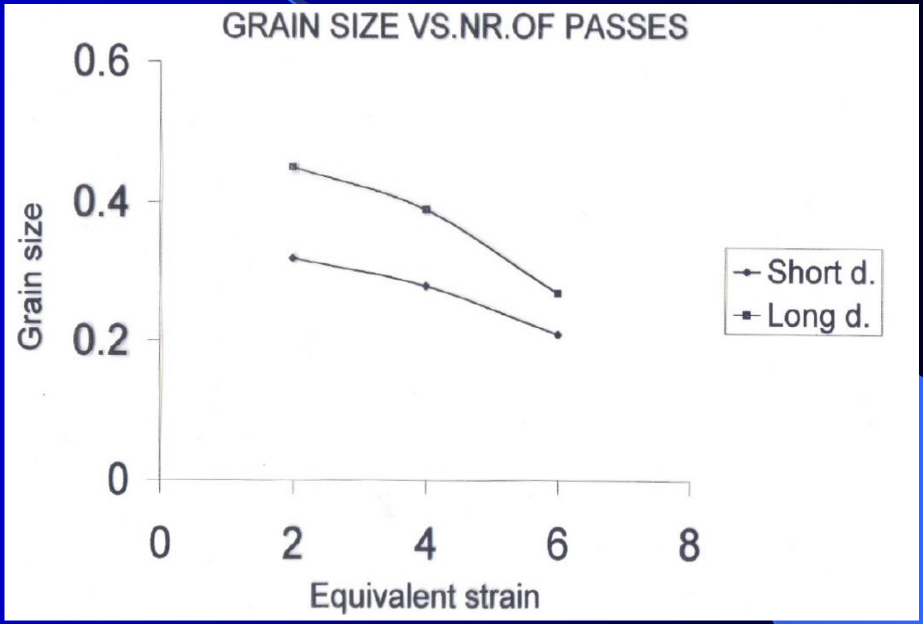
- Microstructure :
- TEM (a Philips CM-200 at 200 kV) ; Specimen preparation: twin-jet polishing

- Microhardness HV
- Tensile testing at room temperature and at a constant rate of 3×10^{-3} s^{-1}

Experimental results

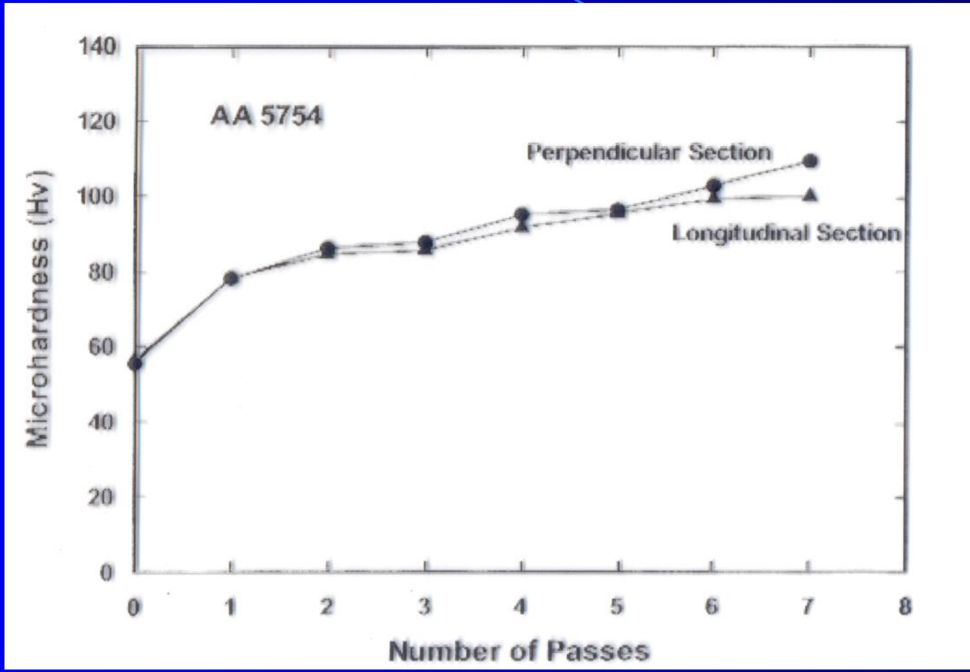
- Microstructure after ECAP through 2 - 6 passes





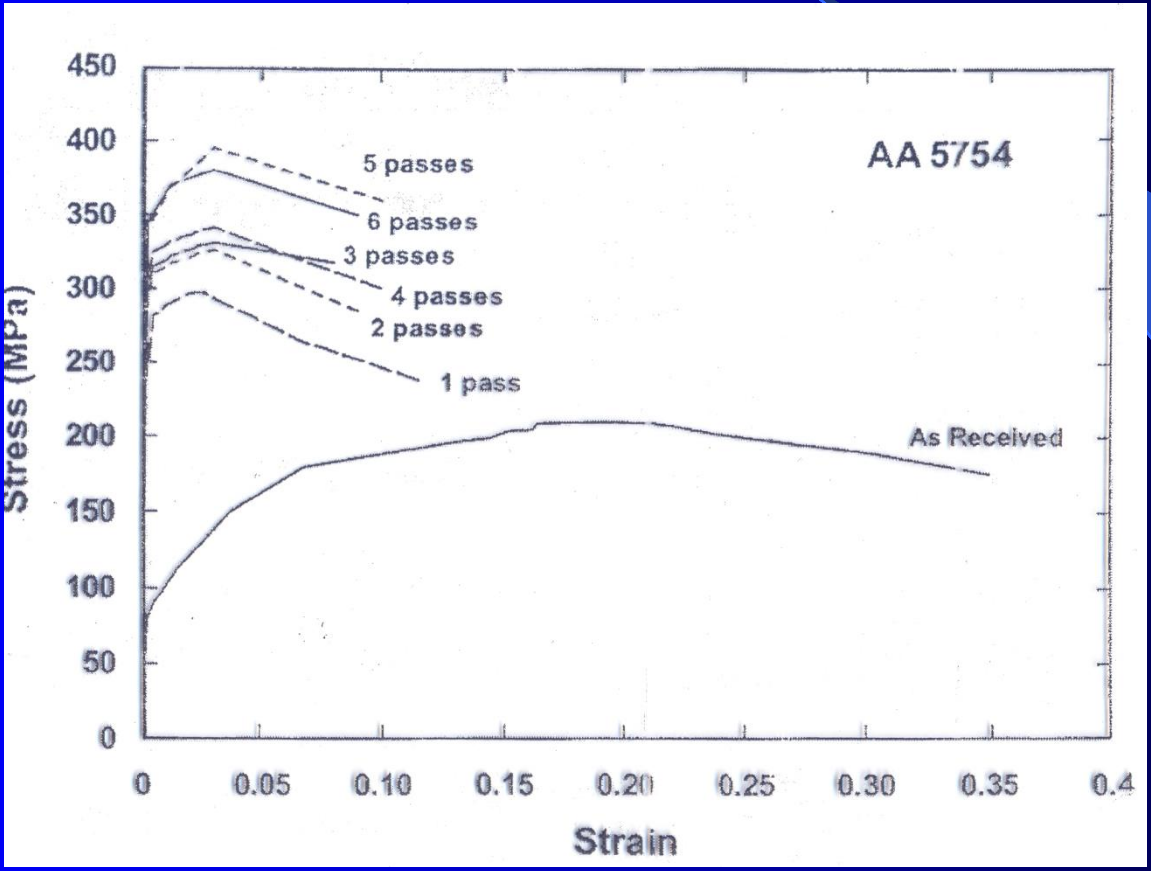
Measurements indicated average grain sizes ~ 0.3-0.4 μm, in the as-pressed condition, demonstrating that ECAP is an effective procedure for attaining an UFG size.

Microhardness *Hv* versus number of passes



- 2 Conclusions:
  - The hardness is essentially independent of the plane of sectioning
  - The value of Hv increases abruptly after a single pass, but thereafter increases slowly with additional passes

Stress vs. strain after pressing through 1 to 6 passes



- The value of 0.2% proof stress increases by a factor ~ 3 times from ~ 80 MPa in the as-received alloy to ~ 240 MPa after a single pass in ECAP
- For additional passes the increase is relatively minor
- Elongations to failure are reduced after ECAP. The unpressed alloy pulls out to elongation ~ 35%, but after pressing from 1-6 passes, the elongation are reduced to ~10%.

Summary and Conclusions

- ECAP was an effective tool for achieving a substantial reduction in the GS of the commercial 5754 Al alloy

- The initial GS of ~ 70 μm in the as received alloy, was reduced to ~ 0.3 - 0.4 μm by ECAP through up to 7 passes.

Cont.

- There is an immediate increase in the microhardness at a strain ~ 1 with minor additional increases with subsequent straining

- 0.2% proof stress is increased by a factor of three times

Strain-rate Sensitivity of (UFG) AA 6061 processed by ECAP

Objective:

- UFG materials may exhibit an enhanced strength and sometimes also an enhanced ductility.
- Investigations on the strain-rate sensitivity of UFG AA 6061 alloy were performed at temperatures ranging from RT to 250^{o} C, in order to reveal the dominant deformation mechanisms.
- The experiment was realized in collaboration with the department of Materials Science and Engineering, Friedrich-Alexander University of Erlangen-Nürnberg, Germany.

Experimental material and procedures

- **AA 6061** commercial alloy with the following chemmical composition in wt% :
0.4-0.8 % Si, 0.7 % Fe, 0.15-0.4 Cu, 0.15 Mn, 0.8-1.2 % Mg, 0.04-0.35 % Cr, 0.25% Zn, 0.15 % Ti with the balance Al.
- The UFG microstructure was achieved by ECAP in a die with an intersecting angle of the die –channels of 120^{o}, using rectangular specimens with a cross section of 16 x 16 mm and a length of 100 mm. For all specimens route B_C was applied.
- For the ECAP process two different initial states of the materials were used.

State 1 : Solution heat treatment at 530^{o} C for 1 hour (quenched in water) + ECAP 6 passes at 100^{0} C

State 2 : Solution heat treatment at 530^{0} C for 1 hour (quenched in water) + 18 hour annealing treatment at 165^{0} C (T6 state) + ECAP 2 passes at 100^{0} C .

•Strain -rate sensitivity (SRS) was determined from strain-rate jumps during compression tests on an Instron 4505 testing machine.

• The samples for the compression tests with the dimensions of d ≈ 4 mm and h ≈ 4.6 – 4.8 mm, were taken from the central part of the ECAP-rods.

• The test temperature was varied from room temperature (25^0 C) up to 250^0 C , which is the upper limit of thermal stability of the UFG-material under the applied testing conditions.

• During the tests the strain rate was varied from 1×10^{-3} s^{-1} to 1×10^{-5} s^{-1}. For the compression test the first and the last applied strain rate was 1×10^{-4} s^{-1}. Thus the testing conditions at the beginning and the end of the complete test series were identical.

Experimental results and Discussion

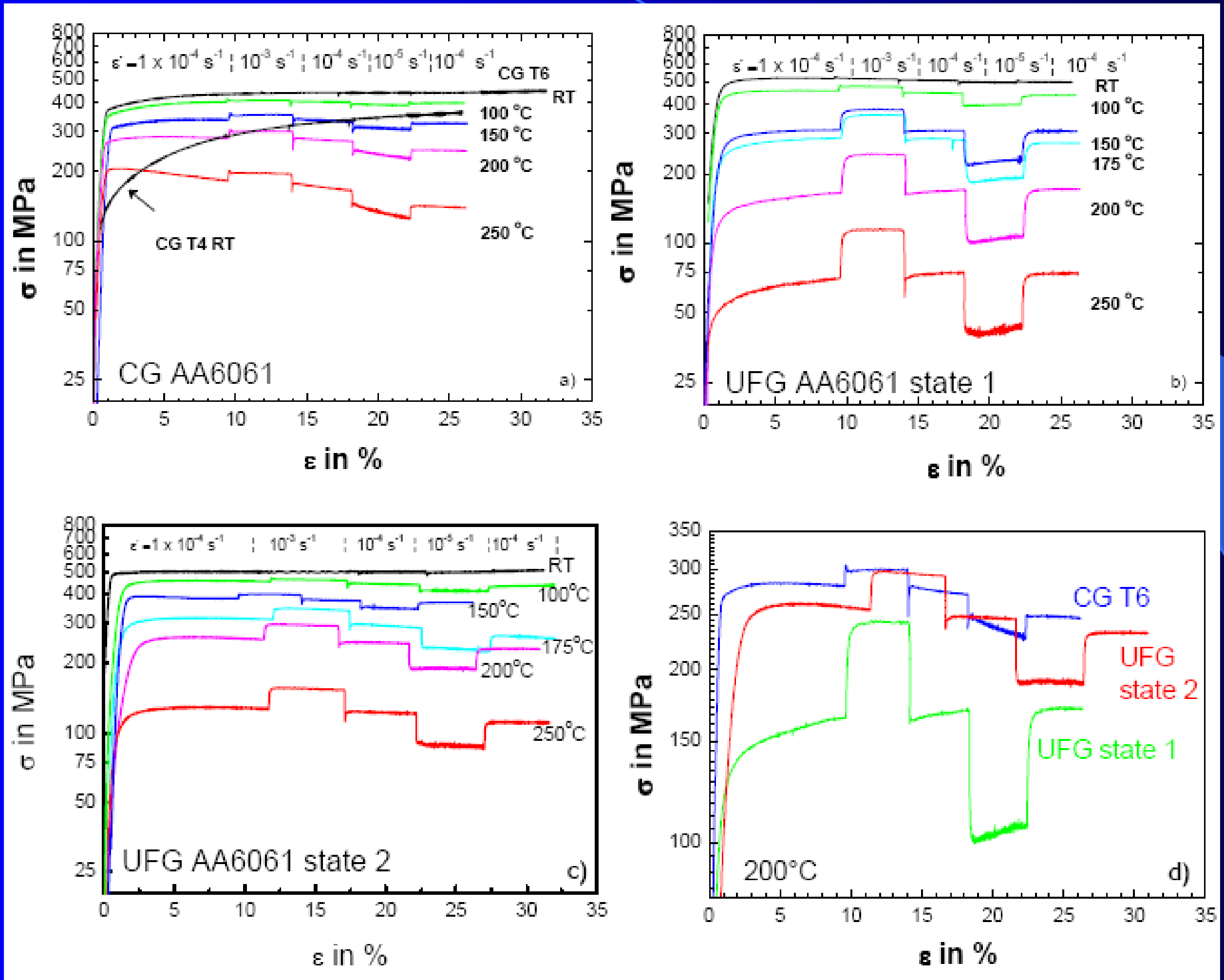


FIGURE 1. True stress – true strain curves from compression tests a) CG AA6061, b) UFG AA6061 state 1, c) UFG AA6061 state 2, d) comparison of the stress – strain curves at 200^0 C for all three conditions. Please note the logarithmic scale of the stress axis.

- Concerning the strain rate sensitivity the following statements can be made:

- For CG AA6061 in a temperature range from room-temperature up to 100^0 C, no pronounced strain rate sensitivity is found. By increasing the testing temperature further the SRS also increases.
- For CG AA6061 only moderate strain rate sensitivity is obtained, compared to the behaviour of both UFG AA6061 conditions.
- For both UFG states the strain rate sensitivity is strongly enhanced compared to the CG condition and the strain rate sensitivity also increases at higher temperatures.

Strain Rate Sensitivity Exponent

- The strain-rate sensitivity exponent *m* was determined by the following formula:

$$m = \left(\frac{\partial \ln \sigma}{\partial \ln \dot{\varepsilon}} \right)$$ where σ is the stress and έ is the strain rate.

- The obtained values of *m* are summarized in the following table

Strain rate sensitivity exponent *m* for CG and UFG AA6061 at different temperatures.

$\dot{\varepsilon} = 10^{-4} - 10^{-3}$ (s^{-1})						
material AA6061	*m* at RT	*m* at 100° C	*m* at 150° C	*m* at 175° C	*m* at 200° C	*m* at 250° C
CG (T6)	-	0.005	0.015	0.026	0.034	0.033
State 2	-	0.01	0.02	0.052	0.068	0.082
State 1	-	0.021	0.096	0.1	0.176	0.22
$\dot{\varepsilon} = 10^{-4} - 10^{-5}$ (s^{-1})						
CG (T6)	-	-	0.019	0.018	0.04	0.046
State 2	-	0.023	0.024	0.058	0.098	0.11
State 1	-	0.05	0.18	0.18	0.24	0.248

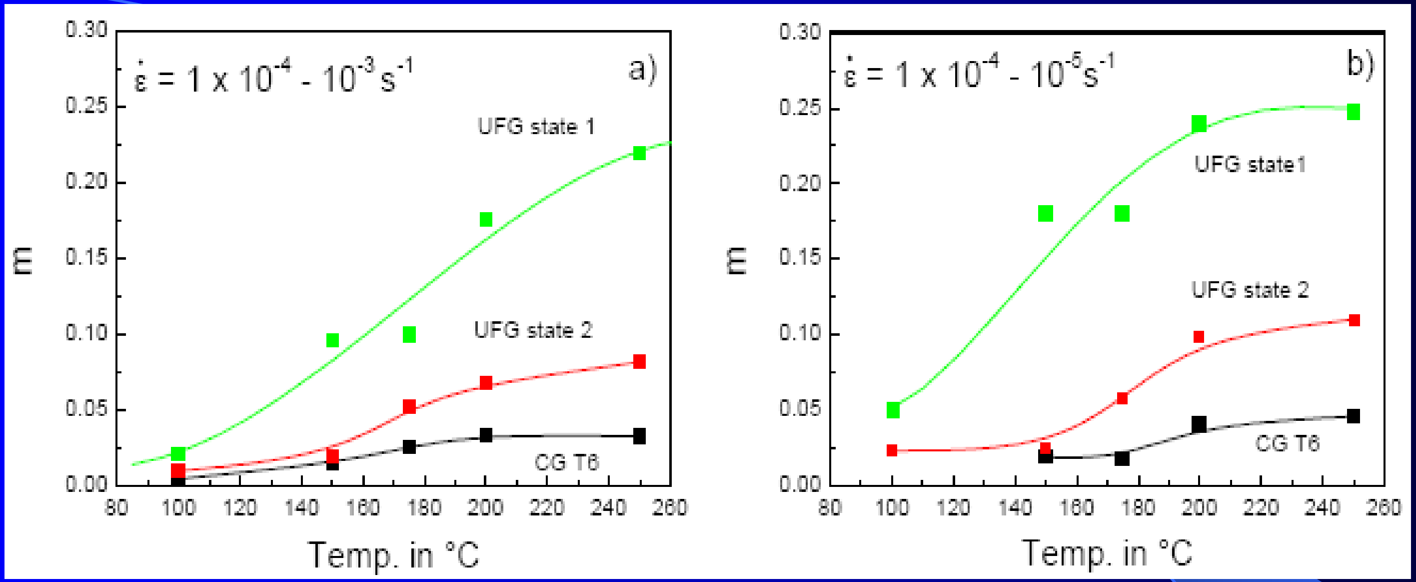


FIGURE 2. Strain rate sensitivity exponent *m* vs. temperature for a) strain rate jumps from 1x 10^{-4} s^{-1} to 1x 10^{-3} s^{-1} and b) 1x 10^{-4} s^{-1} to 1x 10^{-5} s^{-1}.

- The higher amount of high angle grain boundaries for state 1 is supposed to be the main reason for the enhanced SRS.

The results obtained indicate a change in the deformation mechanism in the UFG regime

- It is supposed that thermally activated recovery processes taking place at the grain boundaries are the dominating deformation mechanisms.

Summary and Conclussions

- The SRS of aluminium alloy AA6061, has been investigated in the CG sized state and in the UFG conditions, after ECAP for 2 and 6 passes at 100^0 C.
- Strain rate jumps during compression tests were performed at different temperatures and the values of the strain-rate sensitivity exponent *m* were determined.
- UFG microstructures have a strongly increased SRS compared to CG state, especially at elevated temperatures, indicating a change in deformation mechanism in the UFG regime.
- The value of strain-rate sensitivity *m* increases from *m* = 0.05 at 100^0 C to *m* = 0.248 at 250^0 C (= 10^{-4} – 10^{-5} s^{-1}), for the UFG material processed for 6 ECAP passes
- Thermally activated recovery processes taking place at the GB are the dominating deformation mechanisms.

THANK YOU FOR YOUR ATTENTION