Equal Channel Angular Pressing (ECAP) of hollow profiles made of titanium

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Abstract. Cylindrical tubes made of commercially-pure titanium (CP-Ti) filled with various mandrels (metallic as well as non-metallic materials) were processed by Equal Channel Angular Pressing (ECAP). Different temperatures (500°C down to room temperature), routes (B_c, B_120, C), tools and number of passes (1-6) were applied depending on the mandrel material. For reasons of comparison, solid bolts made of metallic mandrel materials were processed at the same ECAP conditions. The mechanical properties of as-received as well as ECAP tubes and core materials were characterised by hardness mappings (in order to reveal the homogeneity) and tensile tests. The results can be summarised as follows: i) It is feasible to process tubes by ECAP; ii) Using appropriate mandrels, tubes made of CP-Ti can be processed by ECAP at significantly lower temperatures, even at room temperature, as compared to solid bolts; iii) Mechanical properties of tubes and mandrel materials after ECAP are similar to or even better than those of their solid counterparts; iv) Tubes after ECAP at low temperatures show higher strength than what can be achieved in bulk material; v) Excellent homogeneity of microhardness is achieved at least when metallic mandrels and a sufficient number of ECAP passes are used.

1. Introduction

Among the different techniques of Severe Plastic Deformation (SPD) available to obtain bulk nanostructured materials with enhanced mechanical properties, Equal Channel Angular Pressing (ECAP) is the most prominent one, especially when larger sample dimensions are required [1, 2]. However, until now ECAP processing was almost exclusively performed on massive rods and bars, thus, samples with solid (full) cross-sectional area. The main aim of this comprehensive study was to expand the possible cross-sectional shapes of ECAP processed materials and semi-products to tubes (hollow profiles) and to shed more light into the effects of different mandrel materials, processing temperatures, numbers of passes and ECAP die angles on the mechanical properties of tubes and the core materials.

So far, investigations of ECAP of hollow profiles are limited. Moreover, in the great majority of these studies the tubes were not subject of investigations at all but either containers for powder consolidation [1, 3–6] or for encapsulating the target material for using an ECAP die with bigger dimensions [7] or used to protect the core material [8] or both [9]. Nonetheless, some papers deal with the properties of ECAP-processed tubes. Nagasekhar et al. [10] subjected tubes of commercially pure (CP) Ti grade 1 filled with sand to three ECAP passes. The tensile strength increased by approximately 28%, however, the die angle was rather large (150°) and grain refinement was limited. Another interesting study by Al-Mufadi et al. [11] compared polyurethane rubber, sand and grease as inserts into
copper tubes subjected to ECAP. It turned out that rubber and sand worked well as mandrel materials but grease failed. The hardness of the Cu tube increased with increasing number of ECAP passes, though, the minimum grain size achieved after 4 passes at room temperature (RT) was 19 µm. Djavanroodi et al. [12] also investigated copper tubes filled with flexible rubber and showed that ECAP did not change the wall thickness. Additionally, there are investigations on ECAP of Al tubes filled with hydraulic oil [13] and on ECAP of Al as core material in a Cu tube [14–16]. The latter study showed that the strength of Al encapsulated in a Cu tube during ECAP was lower than after conventional ECAP.

2. Experimental procedure

2.1. Materials and samples

Two types of samples were subjected to ECAP: 1) tubes made of CP-Ti grade 2 filled with different (metallic and non-metallic) mandrel materials and 2) solid, cylindrical bolts made of the same materials as the metallic mandrels. Both sample types had the same dimensions: outer diameter of 20 mm and length of 120 mm. The wall thickness of the tubes was 2 mm and thus the inner diameter was 16 mm. Mandrel materials consisted of the metals CP aluminium (hereafter abbreviated to CP-Al), CP-Ti grade 2 (CP-Ti) – nominal the same as the tube material, however, unfortunately with different mechanical properties in the as-received (AR) condition – and the Ti-6Al-4V alloy (Ti64) as well as of two non-metals: graphite (C) and oak wood (oak).

2.2. Equal Channel Angular Pressing (ECAP)

ECAP was performed at AIT Austrian Institute of Technology. Two ECAP tools with a channel diameter of 20 mm and with different angles of intersection (120° and 105°) were used. Routes Bc, B120 and C, in which between subsequent passes the billet is rotated in the same direction by 90°, 120° and 180°, respectively, were applied. Both, tools and samples were held at the same, constant temperature during the whole process, whereby, if needed, the samples were pre-heated prior to the first ECAP pass in an air furnace for 20 minutes. A graphite-based anti-seize paste Molykote® P-74 was used as lubricant.

The critical processing parameters, particularly temperatures and angles of intersection, were adjusted to the mandrel materials and are listed together with routes in table 1. Thus, for the Ti64 core 500°C had to be chosen regardless the fact, that the temperature is too high for processing pure titanium. In general, one of the aims of the study was to conduct ECAP on Ti tubes at the lowest possible temperature. In fact, for CP-Al and non-metallic cores, temperatures as low as 100°C and even RT were used. Further, it was intended to reveal the homogeneity of the microstructure particularly to clarify the question whether the simple shear deformation originating at the point of channels’ intersection can spread through the mandrel, especially the non-metallic ones, and reach the outer section of the sample. Therefore, ECAP with one single pass as well as a higher (optimum) number of passes was performed.

| Table 1. | ECAP parameters for CP-Ti tubes with different mandrel materials. |
|---|---|
| | Mandrel material | Number of passes and route | Temperature [°C] | Angle of intersection [°] | Total uniform equivalent strain acc. [1] |
| Metallic | CP-Al | 1x | 100 | 105 | 0.9 |
| | | 2xC | | | 1.8 |
| | CP-Ti | 1x | 400 | 105 | 0.9 |
| | | 6xB120 | | | 5.3 |
| | Ti64 | 1x | 500 | 120 | 0.6 |
| | | 4xBc | | | 2.5 |
| Non-metallic | C | 1x | RT | 105 | 0.9 |
| | | 1x | 100 | 105 | 0.9 |
| | oak | 2xC | RT | 120 | 1.3 |
| | | 4xCBC | | | 2.5 |

* Rotation sequence: 180°, 90° and 180°
2.3. Mechanical properties

Vickers hardness measurements (HV1) were performed on cross sections perpendicular to the ECAP direction using a fully-automatic hardness tester (EMCO-TEST DuraScan 80) according to EN ISO 6507-1. Hardness mappings on the whole cross section of the tubes as well as of the cores were performed with a mesh of 0.5x0.5mm$^2$ to reveal the respective hardness distributions.

All tensile tests were carried out at RT according to ISO 6892-1 A222 procedure with an initial strain rate of 3x10$^{-4}$ s$^{-1}$. A computerized universal Shimadzu Servopulser EHF-UV050K1 testing machine equipped with a high-precision clip-on extensometer was used. Two types of tensile samples were used. First, thread-end test pieces of round cross-section of type B6x30 according to DIN 50125 obtained by turning from mandrel materials – in as-received (AR) state, as cores of tubes as well as solid bolts after ECAP – were tested. This approach ensured the highest precision of the data. At least two samples per condition were tested in order to single out possible outliers. Second, properties of the tube material – both AR and after ECAP – were obtained on test pieces analogous to DIN 50125 - E2x5x20. Four samples per tube were fabricated to obtain an average value. The two transverse surfaces were machined by spark erosion to tight tolerances whereas the original thickness or the wall remained unmachined.

3. Results

3.1. Hardness and homogeneity

The results of hardness on tubes are shown as colour-coded mappings in table 2 and as numerical data (mean value) in table 5. Processing tubes with a graphite mandrel by ECAP is feasible, but only for one pass. Even so, a vast increase in hardness occurs due to the low deformation temperature (from 180 HV1 in AR condition to 233 HV1 and 217 HV1 after one ECAP pass at 100°C and RT, respectively).

Also in case of tubes with oak mandrel a significant increase in hardness (212 HV1) as well as an inhomogeneous hardness distribution (table 2) after one pass at 100°C were observed. Moreover, several ECAP passes could be performed and resulted in very high hardness (220 HV1 after 2xC at RT and 224 HV1 after 4xCBC at 100°C) as well as in fairly homogenous hardness distributions. However, oak was the only mandrel material which did not preserve the dimensions of the tube during ECAP processing. The tubes partially collapsed and a shortening as well as a nonuniform doubling in wall thickness occurred due to the compressibility of wood.

As CP-Al is a quite soft metallic mandrel material the ECAP temperature can be as low as 100°C. This effectively increased the hardness of the CP-Ti tube, even after one pass, and resulted in a quite uniform hardness distribution on the cross-section of the tube after only 2 passes with route C.

CP-Ti as mandrel material leads to a homogenous hardness distribution as well, though, many passes (6) are needed for an increase in hardness compared to the AR material. This is due to the higher processing temperature of 400°C used for this rather strong mandrel material.

Using mandrels made of Ti64 the ECAP processing temperature had to be even higher (500°C) resulting in rather low hardness values, as a matter of fact, in case of only one ECAP pass even lower than in the AR condition. However, the tensile properties improved (see next chapter).

In case of non-metallic mandrels no investigations of mechanical properties were performed. Hardness results of metallic mandrels represented by colour-coded mappings are shown in table 3 for CP-Ti and in table 4 for Ti64. The mean values are specified in table 5. It can be seen that ECAP of metals as mandrels in CP-Ti tubes leads to higher hardness values and slightly more homogenous hardness distributions (for CP-Ti mandrels) than conventional ECAP processing of solid bolts.
Table 2. Hardness maps of CP-Ti tubes as received (AR) and after indicated number of ECAP passes and routes with different mandrel materials. The top side of the mappings corresponds to the channels’ intersection in the ECAP tools. (For abbreviations see chapter 2.1.)

| Material          | AR                  | C, 1x, 100°C        |
|-------------------|---------------------|---------------------|
| Oak, 1x, 100°C    | Oak, 4xCBC, 100°C   |
| CP-Al, 1x, 100°C  | CP-Al, 2xC, 100°C   |
| CP-Ti, 1x, 400°C  | CP-Ti, 6xB120, 400°C|
| Ti64, 1x, 500°C   | Ti64, 4xBc, 500°C   |

Table 3. Hardness maps of CP-Ti mandrel material AR and after one and six ECAP passes at 400°C as solid bolts and mandrels.

| Material          | AR                  |
|-------------------|---------------------|
| Solid             | Mandrel             |
| 1x                |                     |
| 6xB120            |                     |

Table 4. Hardness maps of Ti64 in as-received state (AR) and after one ECAP pass at 500°C as solid bolt and mandrel.

| Material          | AR                  |
|-------------------|---------------------|
| Solid             | Mandrel             |
| 1x                |                     |
| 3x                |                     |

180 200 220 240
165 190 210 230 255
335 345 355 365 375 385
3.2. Tensile strength and ductility

The numerical results of tensile tests on CP-Ti tubes are summarised in Table 5 while Figure 2a shows stress-strain curves of selected samples. In all cases the yield strength and the ultimate tensile strength (UTS) are increased by ECAP, even at the highest processing temperature of 500°C, which is somehow contrary to the hardness measurements. This is due to a strong texture in the AR condition. The enhancement of the UTS is quite substantial; up to 50% increase is obtained after 6 passes at 400°C with CP-Ti as mandrel or even after one single pass at 100°C with CP-Al as mandrel. A moderate decrease in total elongation is mostly observed after one pass and a further reduction occurs after 6 passes, however, the ductility is still reasonable. Furthermore, a substantial reduction in uniform elongation due to a change in the strain hardening coefficient takes place, which is typical for ECAP materials.

The results of tensile tests on metallic mandrel materials as well as on solid ECAP bolts of the same materials after the same ECAP deformation are summarized in Table 5 and exemplary stress-strain curves are depicted in Figure 2b. In case of CP-Al the improvement of UTS is in the range of 20-25%. The highest increase in tensile strength is obtained for CP-Ti after 6 passes at 400°C (+37%).

Table 5. Mechanical properties of CP-Ti tubes and mandrel materials. The latter were either ECAP-processed as solid bolts (S) or as mandrels of CP-Ti tubes (M). YS denotes yield strength, UTS is ultimate tensile strength, Ag percentage uniform elongation, A percentage elongation after fracture.

| Mandrel material | ECAP conditions | Tubes | ECAP conditions | Tubes |
|------------------|----------------|-------|----------------|-------|
|                  |                | HV1   | YS [MPa] | UTS [MPa] | Ag [%] | A [%] | HV1   | YS [MPa] | UTS [MPa] | Ag [%] | A [%] |
| CP-Al            | AR             | 180   | 289       | 427       | 15     | 27    | 79.5  | 198       | 223       | 7      | 18    |
|                  | 1x, 100°C, 105°| 209   | 543       | 608       | 2.5    | 15    | S     | 90.8      | 269       | 275    | 2.3   | 13    |
|                  | 2xC, 100°C, 105°| 227 | –         | –         | –      | –     | M     | 92.6      | 266       | 276    | 2.8   | 13    |
| CP-Ti            | AR             | 180   | 289       | 427       | 15     | 27    | 202   | 413       | 494       | 11     | 39    |
|                  | 1x, 400°C, 105°| 178   | 432       | 491       | 5.5    | 27.5  | S     | 208       | 527       | 576    | 4.2   | 28    |
|                  | 6xB120, 400°C, 105°| 194 | 507       | 585       | 2.5    | 12.5  | M     | 213       | 547       | 602    | 4.4   | 25    |
| Ti64             | AR             | 180   | 289       | 427       | 15     | 27    | 346   | 977       | 1024      | 6      | 18    |
|                  | 1x, 500°C, 120° | 174   | 422       | 494       | 7.5    | 20    | S     | 356       | 1138      | 1152   | 1.1   | 10    |
|                  |                |       |           |           |        |       | M     | 361       | 1125      | 1146   | 0.9   | 11    |

Figure 2. Tensile stress-strain curves. a) CP-Ti tube b) CP-Ti as mandrel material (M) and solid counterpart (S). Note the different mechanical properties in AR state.
4. Discussion and summary
The current study has proved the feasibility of processing tubular samples by ECAP. By selecting proper mandrel materials it is possible to considerably reduce the pressing force and therefore to lower the ECAP temperature of CP-Ti. This leads to a vast increase in hardness and strength of CP-Ti tubes, even higher than what can be obtained in solid titanium rods by numerous ECAP passes at elevated temperatures. E.g. the highest hardness was reached after only one pass at 100°C with C as a mandrel (233 HV1). However, close attention should be paid to the homogeneity of the microstructure especially when non-metallic mandrels are used in consequence of a non-uniform plastic deformation during processing: The sample area in the vicinity of the channels’ intersection undergoes typical ECAP deformation by simple shear, whereas the opposite site of the tube is simply bent. The most uniform hardness distribution on tube cross-sections were achieved with CP-Ti as mandrel material after 6 passes at 400°C using route Bc. Using mandrel materials with higher strength than the tube material (e.g. Ti64) requires higher ECAP temperatures than necessary for pure titanium resulting in rather low mechanical properties after ECAP. Therefore, soft metallic materials, e.g. pure Al, are recommended for mandrels. CP-Al leads to the highest hardness of the CP-Ti tube compared to all other metallic mandrel materials tested, however, the hardness is lower than in case of C.

Mechanical properties of metals processed by ECAP as mandrels encapsulated in CP-Ti tubes are better (CP-Ti, Ti64) or at least the same (CP-Al) as properties of these materials after conventional ECAP deformation in the form of solid bolts under the same conditions.

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