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# Structure and properties of nanostructured Cobalt processed by high pressure torsion at temperatures of 300 and 77 K

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**Abstract.** Several nanostructured states of high purity cobalt were achieved by high pressure torsion (HPT) at pressures of 4 and 8 GPa and temperatures of 300 and 77 K. Changes of crystallographic texture, grain sizes and phase transformations as a result of HPT and subsequent annealing have been measured and analyzed. Mechanical properties of nanostructured Co were studied by microhardness measurements at 300 K, and uniaxial compression at temperatures 300, 77 and 4.2 K. Comparison of the strength characteristics, plasticity and strain hardening of the nanostructured and coarse grained Co has been carried out. Micromechanisms of plastic deformation of Co during HPT deformation and uniaxial compression are discussed.

## 1. Introduction

In the last years the method of high pressure torsion (HPT), which is one of the most successful methods of severe plastic deformation, was used to produce nanostructured states in hcp metals, such as Ti and Zr [1-5]. HPT at ambient temperature and applied pressure not more than 2 GPa allowed to achieve average grain sizes in Ti and Zr of approximately 160-200 nm [2-5]. The use of cryogenic temperature (77 K) during HPT gives smaller grain sizes (80-100 nm), while applying higher stresses during HPT at both temperatures 77 and 300 K leads to a phase transformation to the  $\omega$  phase. In contrast to Ti and Zr, Co shows no phase transition with increase of pressure, which gives the possibility to apply higher pressures during HPT, resulting in a more stable deformation during HPT and in the perspective to achieve smaller grain sizes with maintaining the hcp crystal structure. On the other hand, deformation twinning is very active at low temperatures in Co in both coarse grained and nanocrystalline state [6]; therefore HPT of Co at a cryogenic temperature of 77 K can lead to the formation of a considerable quantity of deformation twins, which should contribute to strength and plasticity of the nanostructured Co.

Thus, the aim of this work is (i) to produce nanostructured Co by the HPT method at 300 and 77 K, and (ii) to study its structure and properties. As nanocrystalline cobalt with an average grain size 20 nm achieved by electrodeposition method [6] was recently investigated [7], this paper also aims to compare the mechanical properties of nanograined Co in different



structural states: after electrodeposition (low energy grain boundaries) and after severe plastic deformation by HPT (high energy grain boundaries).

## 2. Materials and Methods

Polycrystalline cobalt was chosen as the material of investigation. The average grain size of the initial rod was 5  $\mu\text{m}$ , and the purity of Co was about 99.995 wt.%.

Severe plastic straining of cobalt was performed by High-Pressure Torsion. Disc-shaped billets were cut by spark erosion from the initial Co rod (diameter 6.4 mm) perpendicular to the longitudinal rod axis. The billets were compressed at 575 K to get the disc-shaped samples with diameter 8 mm and thickness 0.77 mm being suitable for HPT. Samples were processed by 5 revolutions of HPT at a hydrostatic pressure of 4 and 8 GPa and temperatures of 300 K and 77 K. The cryogenic temperature of 77 K was achieved by merging both sample and HPT plungers into liquid nitrogen. Shear strains up to about 210 have been achieved through torsion deformation. Values of shear strain have been calculated by using the equation  $\gamma = \pi nr/h$ , with  $n$  as the number of rotations,  $r$  the distance from rotation axis, and  $h$  the sample thickness received after HPT processing. Plungers have been rotated by a speed of 0.2 rot/min. The thickness reduction during HPT processing did not exceed 28 %. Some of the samples were annealed in vacuum at 425, 575 or 975 K for 3 h after HPT deformation.

The microstructure of the HPT samples was studied using a Philips CM200 transmission electron microscope operated at 200 kV. TEM samples were punched out of the HPT discs and electropolished with a polishing solution consisting of 33% nitric acid and 67% methanol at -23°C and a voltage of 4.5 V. Uniformity of HPT deformation was also studied with a Zeiss Supra 55 VP scanning electron microscope in EBSD mode. Samples for SEM were mechanically polished and electropolished with 85% phosphoric acid.

The X-ray diffraction measurements were performed in transmission at the High Energy Materials Science Beamline at PETRA III/HASYLAB using monochromatic radiation with 50 keV energy. The beam size was  $200 \times 400 \mu\text{m}^2$  and the scattered intensity of 12 reflections was recorded with a Perkin Elmer XRD 1622 Digital X-ray Flat Panel Detector. The 2-D data were azimuthally integrated by FIT2D software yielding intensity vs.  $2\theta$  line profiles.

Texture measurements of coarse grained and HPT processed Co were performed by means of a high resolution Bruker-AXS D 8 X-Ray diffractometer in combination with a 2D detector Bruker HISTAR, using X-ray Fe radiation. Textures were measured in the plane, which is perpendicular to the radius of the HPT (and initial coarse grained) disc, see Fig. 1. Pole figures  $(10\bar{1}0)$ ,  $(0002)$ ,  $(10\bar{1}1)$ , and  $(10\bar{1}2)$  have been registered. The Orientation Distribution Function (ODF) was calculated using the Arbitrary Defined Cells (ADC) method [8] being part of LABOTEX 3.0 texture analysis software [9]. The pole figures shown have been recalculated from the ODFs.

To measure the Vickers microhardness, HPT discs were highly polished and the microhardness was measured with an applied load of 1.5 N for 15 s along the radii from the center to edge at 4 different radial directions, in steps of 0.5 mm.

Compression samples with the shape of rectangular prisms  $0.6 \times 0.8 \times 1 \text{ mm}$  were cut from HPT processed discs and coarse grained initial material discs by spark erosion in such a way (Fig. 1) that the largest dimension of the sample was along the radial direction of the HPT disc, which was also chosen as the compression axis. The samples were cut from the layer 1-3 mm from the center of HPT discs.

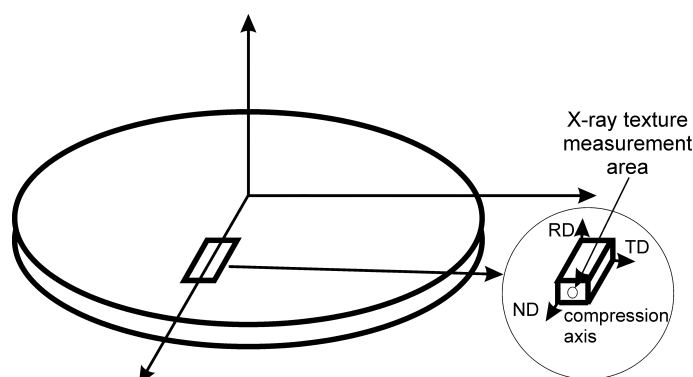


Fig. 1. Sketch of cutting the compression samples from HPT or initial coarse grained discs. The insert on the right shows the compression sample with the direction of compression axis and reference system of the texture pole figures

The mechanical characteristics of nanostructured and coarse grained Co samples were studied at temperatures of 300 K, 77 K (in liquid nitrogen), and 4.2 K (in liquid helium) by recording the load-time curves for compression of the sample with a relative strain rate of  $5 \cdot 10^{-4} \text{ s}^{-1}$  in an MRK-3 deformation machine. The registered curves were converted into stress-plastic strain diagrams  $\sigma(\epsilon)$ . The stress quantity  $\sigma$  was determined as the ratio of the load to the initial cross-sectional area of a sample, and the value of plastic strain  $\epsilon$  was calculated as the ratio of the change of the length of a sample due to plastic strain to its initial length.

### 3. Results and Discussion

#### 3.1. Electron microscopy

Scanning electron microscopy in EBSD mode of the nanostructured Co after HPT deformation at temperatures of 300 and 77 K has shown that the grain size distribution in longitudinal and transversal sections of the HPT discs is rather uniform. The samples show a nanograined microstructure.

A more detailed study of the microstructure by TEM has shown the presence of a high concentration of dislocations in grains after HPT at 77 K (Fig. 2a). Internal stresses are very high, grain boundaries are wide and indicate high-energy dislocation arrangements.

The average grain size of the nanostructured Co (HPT at 77 K) is 70 nm (Fig. 3a). This value is in good correlation with other nanostructured hcp metals processed in similar conditions: Ti (100 nm) [5] and Zr (80) nm [4]. A special feature of this cryogenic processing is the high activity of twinning [10].

HPT at a temperature of 300 K also led to a severely deformed microstructure (Fig. 2c), but a considerable number of recrystallized nanosized grains is observed here due to a higher efficiency of the dynamic recovery at this processing temperature [11, 12]. The average grain size after HPT at 300 K is 100 nm (Fig. 3c).

Annealing in vacuum at a temperature of 425 K of nanostructured Co after HPT processing at both temperatures 300 and 77 K (Fig. 2) does not lead to a considerable change of the grain size distribution (Fig. 3), and average grain sizes of 110 and 80 nm result, respectively. After annealing the level of internal stresses and the density of dislocations has been decreased.

Annealing of the nanostructured Co at higher temperature (575 K) leads to significant grain growth.



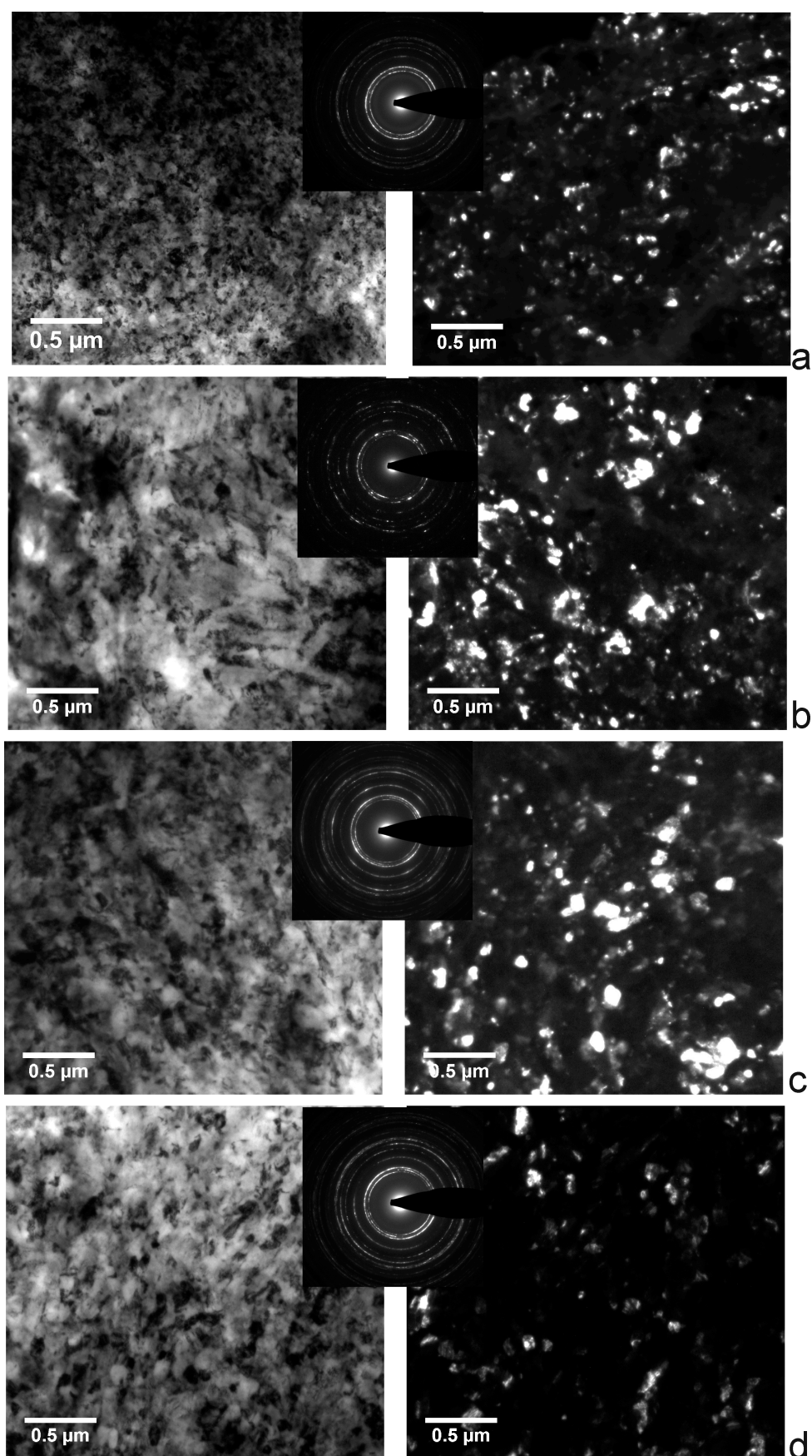


Fig. 2. Bright field TEM (left column), dark field TEM (right column) and SAED patterns (centers) of the nanostructured Co, processed by HPT (8 GPa, 5 rotations) at temperatures 77 K (a); 77 K + annealing at 425 K (b); 300 K (c); 300 K + annealing at 425 K (d).

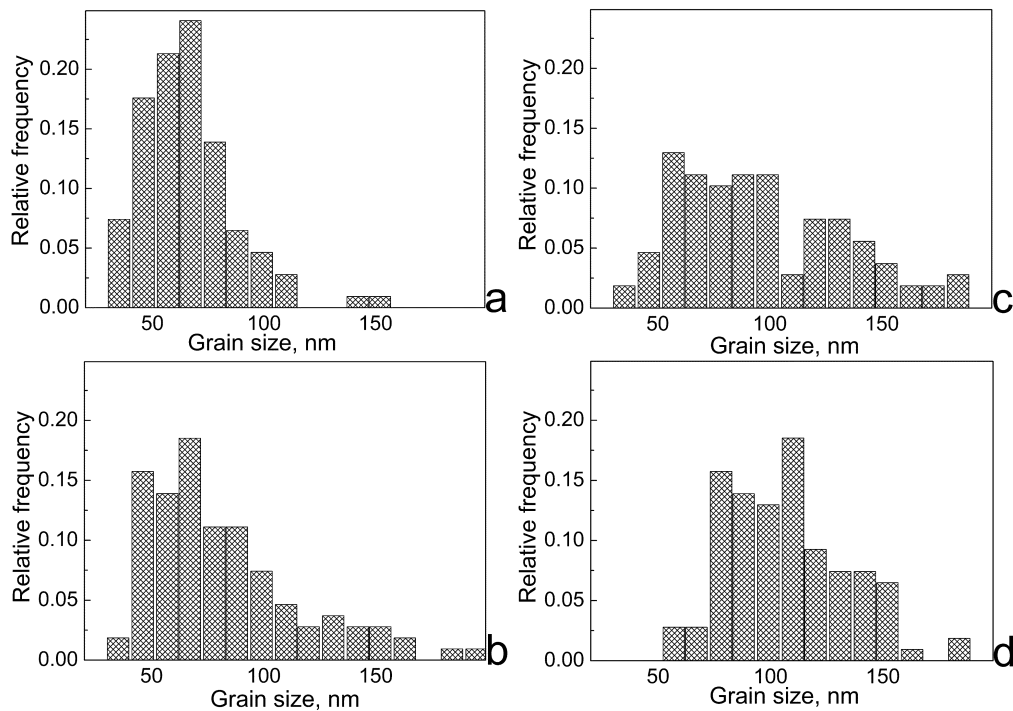


Fig. 3. Grain size distributions of the nanostructured Co, processed by HPT (8 GPa, 5 rotations) at temperature 77 K (a); 77 K + annealing at 425 K (b); 300 K (c); 300 K + annealing at 425 K (d).

### 3.2. X-ray analysis

Fig. 4 shows X-ray diffraction of Co in the initial coarse grained state, in the nanostructured state (after HPT) and after additional annealing.

It can be seen from Fig. 4 that initial coarse grained Co exhibits the hcp  $\alpha$  phase, which is typical of this material at ambient conditions. HPT at 8 GPa leads to a change of the  $\alpha$  peaks intensity due to changes of texture during HPT deformation, but no phase transitions occur. Annealing at a temperature of 425 K does not lead to a phase transition either, whereas annealing at 975 K leads to a nearly complete transition to the fcc phase and after cooling the quasistable two-phase structural state is formed (Fig. 4). Such a phase transition during heating (approximately at 700 K) was registered earlier for coarse grained Co [13].

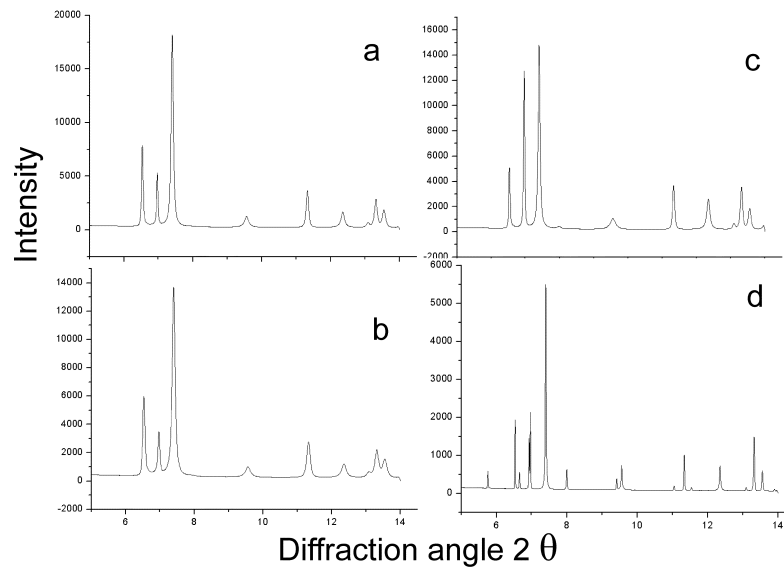


Fig. 4. X-ray diffraction of Co in initial coarse grained state (a), nanostructured Co (HPT at 8 GPa, 300 K) (b), nanostructured Co (HPT at 8 GPa, 300 K) + annealing at 425 K (c), nanostructured Co (HPT at 8 GPa, 300 K) + annealing at 975 K (d).

The crystallographic texture of Co in initial coarse grained and nanostructured states is shown in Fig. 5.

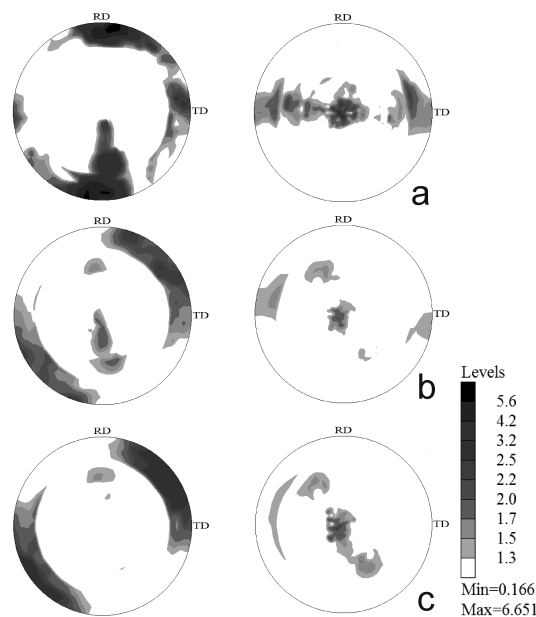


Fig. 5. (0002) and  $(10\bar{1}0)$  pole figure (left and right column) of Co in initial coarse grained state (a), nanostructured Co (HPT at 8 GPa, 300 K) (b), nanostructured Co (HPT at 8 GPa, 77 K) (c).

In Fig. 5 the radial direction (RD) is perpendicular to the plane of the HPT disc (and the disc of coarse grained Co), the normal direction (ND) corresponds to the radius of HPT disc (see Fig. 1 for more details). (0002) and  $(10\bar{1}0)$  pole figures demonstrate the distribution of basal planes and prismatic planes of crystallites, respectively. Fig. 5 shows that HPT deformation leads to a significant change of texture – the  $c$  axis of crystallites (which is

normal to the basal plane) of a majority of crystallites acquires an orientation approximately  $90^\circ$  to the normal direction (ND). A large part of crystallites has nearly uniformly distributed between the RD and of the TD direction. Such a distribution of crystallite orientations as a result of HPT is typical of an hcp metal with a preferable activity of basal dislocations, such as Mg [14]. So, taking into account that the motion of basal dislocations is the main deformation mechanism of coarse grained Co at low homological temperatures [15], it can be suggested that the main contribution to plastic deformation during HPT consists of basal dislocations.

The (0002) pole figure of nanocrystalline Co (HPT at 300 K) (Fig. 5b) demonstrates also several maxima in a direction, which is normal to TD, indicating a contribution of prismatic and pyramidal dislocations to plastic deformation during HPT [16]. During severe plastic deformation at cryogenic temperature (Fig. 5c), the intensity of these maxima and correspondingly the activity of prismatic and pyramidal dislocations is much lower.

### 3.3. Mechanical properties

It can be seen from Table 1 that HPT deformation leads to a considerable increase of Co microhardness. The main reason for that is a decrease of the average grain size due to HPT: HPT at 77 K gives a nanostructured state with grain sizes of 70 nm, which is almost two orders of magnitude smaller than the initial coarse grained Co (5  $\mu\text{m}$ ). This leads to a twofold increase of the microhardness of the nanostructured Co in comparison with the coarse grained material. HPT at 300 K gives larger grain sizes (100 nm) and thus the microhardness of this nanostructured state is approximately 10 % smaller than for HPT at 77 K. Such a difference in strength (10-15%) for structural states produced by severe plastic deformation at 300 and 77 K is also typical of other hcp metals (Ti and Zr) [5, 17]. HPT of Co at 4 GPa give a similar microstructure and hardness as in the case of HPT processed at 8 GPa indicating that both these pressures achieve the same grain refinement and strength as some saturation may have been reached already with a value of 4 GPa.

Table 1. Microhardness of initial coarse grained and nanostructured Co (after HPT deformation at 8 GPa and annealing at different temperatures).

	coarse grained	HPT at 77 K	HPT at 300 K	HPT at 77 K + annealing at 425 K	HPT at 300 K + annealing at 425 K	HPT at 77 K + annealing at 575 K
Vickers Micro-hardness, GPa	2.30	4.50	4.17	4.42	4.20	2.21

Annealing of nanostructured Co at a temperature of 425 K does not lead to a considerable change of microhardness, while an increase of the annealing temperature to 575 K leads to a significant decrease of the microhardness down to values typical for the coarse grained structural state. Scanning electron microscopy also reveals considerable recrystallization in these structural states – some grains show sizes of several micrometers. Such a behavior is different from the electrodeposited nanocrystalline Co with 20 nm average grain size, where no decrease of strength is registered after annealing at temperatures up to 673 K [6]. This difference can be explained by a higher concentration of impurities (especially sulfur), which is typical of electrodeposited materials. Atoms of sulfur can be efficient barriers for the carriers of plasticity (dislocations) and also for motion of grain boundaries, so grain growth is expected to start at higher temperatures in material with a higher concentration of impurities. In the present work high purity Co is studied and its recrystallization temperature is correspondingly lower in comparison with electrodeposited Co. A similar behavior of

thermal stability of strength was observed for other low and high purity hcp metals, e.g. Ti [5].

The yield strength of nanostructured and coarse grained Co (Fig. 6) demonstrates the same effects as hardness (Table 1): the yield strength of nanostructured Co (HPT at 300 K, 100 nm) is approximately two times higher than the one of coarse grained Co. A decrease of the grain size down to 70 nm (HPT at 77 K) leads to an additional increase of 10% in strength at all studied temperatures. As can be seen from Fig. 6, strength at temperature 77 K of HPT deformed and annealed structural states is 15-20 % higher in comparison with not annealed states, whereas at the other temperatures that were studied (300 and 4.2 K), this effect is less pronounced.

This effect may be explained, for example, by the different level/distribution of internal stress in as-HPT processed and additionally annealed samples: in some places of un-annealed nanostructured Co, the high level of internal stresses favours the dislocations to pile off at a comparably low yield strength; in annealed states internal stresses are reduced and a higher applied stress is needed to initiate dislocation motion. At 77 K, the number of active slip systems is smaller than that at 300 K in hcp metals [5], so the stress required to start yielding without action of internal stresses is even higher.

A decrease of compression temperature from 300 to 4.2 K leads to an increase of strength at 40-60% for all structural states studied, indicating that thermoactivated plastic deformation occurs. Barriers for the thermoactivated motion of carriers of the plasticity (dislocations) can consist of linear defects inside the grains in the case of coarse grained Co and peculiarities at grain boundaries in the case of nanostructured Co.

The strength of nanostructured Co, processed by HPT, (Fig. 6) can be compared with electrodeposited Co [6]: for strain rates  $10^{-4}$ - $10^{-3}$  s $^{-1}$  the yield strength  $\sigma_{0.2}$  of the electrodeposited Co (average grain size 20 nm) is 1.04 GPa at 300 K and 1.73 GPa at 77 K [6]; the yield strength of the nanostructured Co (HPT at 77 K + annealing at 425 K, average grain size 80 nm) is 0.93 GPa at 300 K and 1.29 GPa at 77 K. These materials differ in grain size by a factor of four (80 and 20 nm) and only by 10-35 % in strength. This difference can be not only due to grain size, but also because of lower purity of electrodeposited Co – it is known that an increase of impurity concentration can drastically enhance the strength of hcp metals [18]. So, the electrodeposition method allows producing nanocrystalline Co with smaller grain sizes in comparison with the HPT method, but does not necessarily lead to a significant difference in strength. This is probably due to a weakening of the Hall Petch effect typical of nanomaterials with grain sizes below 100 nm [19].

In compression experiments samples of Co were deformed until failure, so the plasticity of nanostructured and coarse grained Co can be analyzed (Table 2). At a temperature of 300 K, the best plasticity is shown by coarse grained Co. HPT deformation leads to a considerable decrease of plasticity due to a significant presence of possible crack nucleation sites (stress concentrators, dislocation pile-ups, etc.) in these severely deformed materials. Annealing of the HPT deformed samples leads to a decrease of internal stresses and to an increase of plasticity, which is only a little smaller than the plasticity of initial coarse grained Co. Comparing the plasticity at 300 K of the electrodeposited Co [6] – 9.5 % of plastic deformation until failure - with plasticity of HPT deformed and annealed Co (Table 2) – 10.2 % - it should be noted that both these materials exhibit a nanograined structure with a rather low level of internal stresses, and despite the entirely different production method, the plasticity (as well as the strength) of both materials is rather similar.

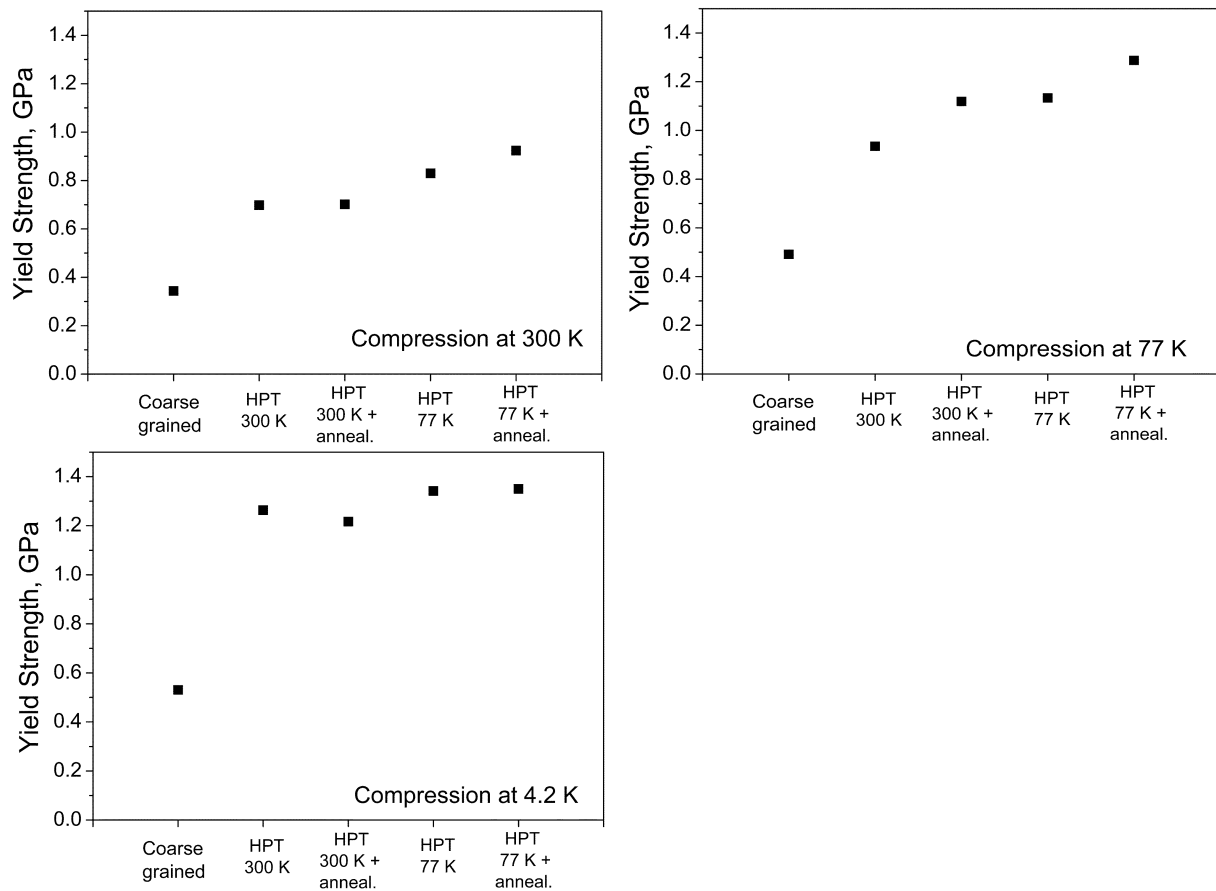


Fig. 6. Yield strength  $\sigma_{0.2}$  of nanostructured and coarse grained Co measured in compression at temperatures 300, 77 and 4.2 K.

A decrease of the compression temperature down to 77 K leads to a decrease of plasticity for all studied structural states, which can be explained by taking into account that in hcp metals at cryogenic temperatures fewer slip systems are active [20]. Therefore, slip of basal dislocations (main deformation mechanism of Co [15]) should represent the overwhelming part of deformation in Co at 77 K, but this deformation mechanism alone does not give a sufficient number of independent slip systems to enable the compatibility of deformation in all grains. This in turn increases the probability of stress concentration and failure of the material at earlier stages of deformation. The decrease of plasticity at 77 K in nanostructured Co is more pronounced in comparison with the coarse grained structural state as the compatibility of deformation in nanosized grains has additional difficulties. The plasticity of Co in all structural states studied is higher at 4.2 K in comparison with 77 K, which may be explained by possible activation of quantum effects in motion of dislocations at such a low temperature [21].

Table 2. Plasticity of initial coarse grained and nanostructured Co (after HPT deformation and annealing).

Temperature, K	Plasticity, %				
	coarse grained	HPT at 77 K	HPT at 300 K	HPT at 77 K + annealing at 425 K	HPT at 300 K + annealing at 425 K
300	13.8	4.1	2.7	8.8	10.2
77	8.8	1.2	1.0	1.3	1.4
4.2	17.6	1.7	2.4	3.0	3.4

The peculiarities of the stress – plastic strain curves can be observed in Fig. 7 for the case of compression at 300 K, where all structural states studied demonstrate considerable plasticity.

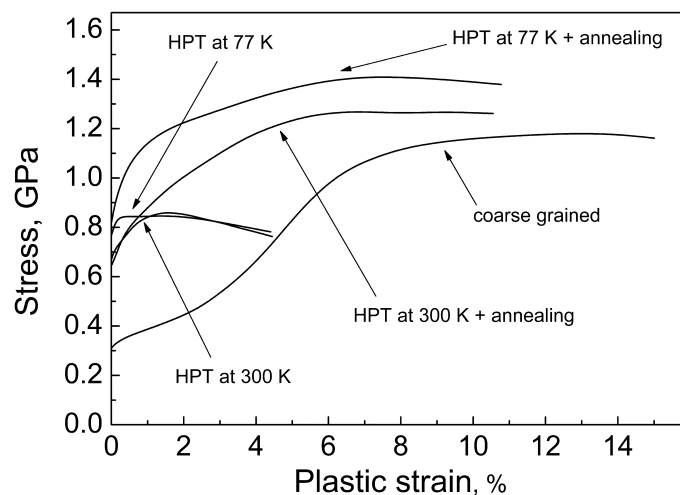


Fig. 7. Deformation curves in compression at temperature 300 K of nanostructured (after HPT and annealing) and coarse grained Co.

Initial coarse grained Co demonstrates several stages of plastic deformation (Fig. 7): an initial parabolic stage – up to approximately 0.5 % of plastic strain; a steady, nearly linear stage – up to 3 % of strain; a significant increase of strain hardening – up to 8 % of strain; nearly easy glide until failure. This type of deformation curve is also typical of other coarse grained hcp metals [22, 23], and the observed significant increase of strain hardening at strains of 3-8 % can be explained by action of twinning: it is known that twinning is active in Co even at ambient temperatures [24] and that twin boundaries can be efficient barriers for dislocations, which leads to an increase of strain hardening [22]. Nanostructured Co after HPT at 77 and 300 K demonstrates the initial parabolic stage (up to 2 %) – Fig. 7, which is followed by decrease of strength with increase of plastic deformation. This loss of stability of the samples is probably caused by accumulation of structural defects during HPT deformation like dislocation pile-ups further leading to nanocracks, etc. Annealing of the HPT nanostructured Co seems to remove those artefacts, and the deformation curves of annealed nanostructured Co show a rather simple shape (Fig. 7) with an extended parabolic-

type initial stage (up to several percents of plastic deformation), which is followed by stable stage of nearly easy slip. The initial parabolic stage is typical of many nanograined materials and is associated with a gradual spread of deformation from bigger grains down to smallest nanosized grains [25]. The stage of parabolic hardening stops when all the grains are involved in the process of plastic deformation. A similar shape of deformation curves was registered for electrodeposited nanocrystalline Co [6]. During plastic deformation of the nanostructured Co, twinning can also be active, but its effect on the shape of the deformation curves cannot be unambiguously identified, and additional structural studies are planned to clarify this question.

#### 4. Conclusions

- A nanostructured state with an average grain size around 100 nm was produced by HPT of Co at 300 K. Cryogenic HPT at 77 K allows to get smaller grain sizes (70 nm) due to reduced dynamic recovery and higher twinning activity at low temperatures;
- Annealing at 425 K does not lead to a considerable change of grain size of the nanostructured Co, while annealing at 575 K leads to significant recrystallization and grain growth. Annealing at 975 K leads to a transition to the fcc phase and to a quasi-stable two-phase structural state after cooling;
- The analysis of crystallographic textures of initial coarse grained and nanostructured HPT Co indicates preferable activity of basal slip systems/dislocations during HPT deformation.
- The strength of the nanostructured HPT Co is up to 2.7 times larger than that of coarse grained Co, and is comparable with the strength of nanocrystalline electrodeposited Co although its grain size is markedly larger. The reason for this result may arise from the decrease of Hall-Petch slope with grain sizes smaller than 100nm as well as from the different purities of materials compared.

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