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Method for slippage evaluation at various stages of high-pressure torsion and its application to Fe-0.1% C

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For materials such as copper and mild steel Fe-0.1% C, slippage has been determined at various stages of high-pressure torsion, and a new effective method has been applied. It has been shown that slippage at the initial stages of high-pressure torsion is insignificant, while after high-pressure torsion for $n=5$ revolutions, torsional deformation of the steel does not occur due to slippage. Transmission electron microscopy and X-ray diffractometry were performed to reveal the microstructure of Fe-0.1% C after high-pressure torsion with $n=5$, 10 revolutions. It has been shown that irrespective of slippage, the structure of Fe-0.1% C is still refined during high-pressure torsion for $n=5$, and a nanostructured state similar to that observed by other authors is formed after HPT in Fe-0.1% C. The data of TEM, XRD and HV unambiguously indicate, that the structure after HPT $n=10$ is more refined and riveted than after HPT $n=5$. Hence, despite the slip at HPT when $n\geq 5$, the deformation is still carried out. One of the possible explanations for the accumulation of deformation in the sample during HPT, despite the slippage, may be that the planes of the upper and lower anvils are declined from each other by a small angle. This leads to the accumulation of significant deformation during the rotation of the anvils.

Keywords: high-pressure torsion, sliding effect, strain, steel Fe-0.1% C.

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Метод оценки проскальзывания на различных стадиях интенсивной пластической деформации кручением и его применение на примере низкоуглеродистой стали Fe-0.1% C

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При помощи нового эффективного метода определено проскальзывание на различных стадиях интенсивной пластической деформации кручением для таких материалов, как медь и низкоуглеродистая сталь Fe-0.1% C. Показано, что на начальных этапах деформирования проскальзывание незначительно, а после ИПДК на $n=5$ оборотов деформация кручением не происходит из-за проскальзывания. Проведены исследования структуры стали Fe-0.1% C после ИПДК на $n=5$ и 10 оборотов методами просвечивающей электронной микроскопии и рентгеновской дифрактометрии. Было показано, что, несмотря на проскальзывание, структура стали Fe-0.1% C все ещё измельчается после ИПДК на $n=5$ оборотов, и в ней формируется наноструктурированное состояние, подобное наблюдаемому другими авторами для аналогичных материалов. Данные ПЭМ, РСА и измерения микротвердости однозначно указывают на то, что структура после ИПДК $n=10$ измельчена и наклёпана сильнее, чем после ИПДК $n=5$. Следовательно, несмотря на проскальзывание при ИПДК, деформация все же осуществляется. Одним из возможных объяснений накопления деформации в образце может быть то, что плоскости верхнего и нижнего бойка отклонены друг от друга на небольшой угол. Это приводит к накоплению значительной деформации при вращении бойков.

Ключевые слова: интенсивная пластическая деформация кручением, проскальзывание, деформация, сталь Fe-0.1% C.

1. Introduction

High-pressure torsion (HPT) is a common method for transforming the structure, forming a nanostructural state, and improving the mechanical properties of metallic materials [1–3]. The HPT method allows you to achieve maximum grain refinement and to determine the effect of large deformations on phase transformations, which is the subject of many articles and a number of reviews [1–3].

For HPT, specimens in the form of thin discs are placed on anvils made of hard material, a pressure of 5–6 GPa is applied, one of the anvils rotates, and surface friction forces lead to shear straining. The material undergoes deformation during hydrostatic compression under the applied pressure from the external layers of the specimen, which prevents the specimen from destruction, despite the high rates of plastic strain. When using the HPT method, parameters can be varied: applied pressure, number of revolutions, strain temperature, anvil rotation speed, anvil geometry, initial state of the deformed material, content of impurities, etc. [1].

In pure metals, HPT leads to the formation of a nanostructured state. For instance, in specimens of Armco-Fe [4] and Ti [5], a grain size of about 80 nm was achieved during HPT. After HPT, a nano-sized honeycomb structure with individual heteraxial nano-sized grains was formed in the steel with the composition Fe-0.1% C, the average size of an element of the grain-subgrain structure was 100–200 nm [6,7]. The initial laminar cementite was dissolved and ultra-fine carbides were produced.

The degree of shear strain γ at HPT is evaluated using the formula

$$\gamma = \frac{2\pi R n}{h}, \qquad (1)$$

where n is the number of revolutions, R is the radius from the center to the measurement point, h is the specimen thickness.

As some studies show, in particular [1,3,8–11], the actually achieved strain γ during HPT of hard or hardened metals and alloys can be much lower than the expected one, which is predicted by formula (1). This inconsistency can be explained by the slippage effect of anvils over the specimen surface during HPT of hard and strain-hardened materials [3]. Starting with the first publication of Bridgeman [8], slippage due to insufficient friction force between the anvils and the specimen is an important issue. The extent of slippage depends critically upon the material and pressure [9]. In the mentioned article, parallel marks were applied to the top and bottom surfaces of the original disc to assess slippage. When aluminum is deformed, very little slippage is observed, slightly more when copper is deformed, and when iron is deformed, slippage becomes significant [9]. The significance of slippage increases both at faster rotational speeds and at lower pressures [9,12]. However, it was not possible to estimate the degree of deformation at HPT by an $\theta > 90°$, since the applied lines were "erased" from the surface of the disks.

It is known that for well-hardened materials there is a certain critical pressure (P) and a degree of deformation at which the material is hardened so much that shear strain at HPT no longer increases due to the slippage effect [3]. However, techniques for detecting slippage by determining torsional torque, marking lines or scratching the sample surface may not be effective [3,9]. In [11], a new convenient method was proposed for assessing the degree of slip/real deformation at HPT, and it was shown that in hard bulk metallic glasses (BMG) based on Zr with tensile yield stress over 1500 MPa, the shear strain is hundreds times less than expected by formula (1) for HPT $n = 0.25–5$. At the same time, despite the slippage, there was a significant change in the structure of BMG after HPT, similar to those observed by other authors during HPT of similar BMGs. In [13], studies of slippage during HPT of the Zr-1% Nb alloy were carried out. It was shown that if the specimen of alloy Zr-1% Nb at initial HPT stages received a significant degree of shear strain, then after HPT with $n > 5$ revolutions, high slippage started and the shear strain does not occur. At the same time, the structure and properties of the Zr-1% Nb alloy change after HPT similar to what other authors observed in such alloys. However, this technique has hardly been used to assess slip during deformation of crystalline metallic materials. The purpose of this work is to determine the degree of shear strain achieved, in particular, on Cu and Fe-0.1% C with an increase in the degree of HPT deformation.

2. Materials and methods

For the study, we selected commercially pure copper and mild low-carbon steel Fe-0.1% C. The original billets were cut according to the diameter of the HPT anvils. For HPT of copper, anvils 20 mm in diameter with a groove 0.5 mm deep were used, and for HPT of steel, anvils 10 mm in diameter with a groove 0.3 mm deep were used. HPT was carried out at room temperature (RT) at 6 GPa. To determine the degree of deformation using the method described in [11] (see Fig. 1a, b, in mentioned article), some of the obtained discs were preliminarily cut into two half-segments, the edges of the segments were polished and varnished to avoid metal adhesion. The segments were placed on anvils according to the scheme in Fig. 1a and subjected to simultaneous HPT. In addition, solid Fe-0.1% C discs were subjected to HPT for $n = 5$ and 10 revolutions at $P = 6$ GPa at room temperature. The microhardness of the obtained steel samples was measured at a load of 1 N applied for 10 seconds. The structure of the samples was studied using a JEOL JEM-2100 transmission electron microscope (TEM). X-ray diffraction (XRD) analysis was carried out on a Rigaku Ultima IV diffractometer using $Cu_{K\alpha}$-radiation.

3. Results

To determine the amount of slippage, 4 series of experiments were carried out.

Experiment I. To determine the actually achievable degree of deformation, the original copper disc was cut into two halves/segments. Then these segments were subjected to joint HPT with a rotation angle of the anvils $\theta = 90°$ ($n = 1/4$) according to the scheme in Fig. 1a. The relative displacement of the edges of the segments as a result of HPT shows that the appearance of the copper segments corresponds to deformation by rotation by $\theta = 90°$ (Fig. 1b).

Experiment II. First, a full copper disc was subjected to HPT for $n = 5$ revolutions, then the resulting HPT disc was cut (Fig. 1c) and the segments were subjected to joint HPT for $\theta = 90°$ (Fig. 1d). The sample received a noticeable deformation by torsion, but there is no significant displacement of the upper part of the segment relative to the lower one (Fig. 1d). Thus, after HPT for $n > 5$, some slippage is observed even on Cu. The degree of deformation is difficult to assess due to the complex geometry of the samples.

Experiment III — Segments of the initial low-carbon steel Fe-0.1%C subjected to HPT $\theta = 90°$ (Fig. 1e). The view of the segments with the edges displaced relative to each other shows that as a result of HPT, the steel sample underwent a noticeable shear deformation. The real shear strain γ_{real} is calculated by the formula $\gamma_{real} = \Delta x/h$, where Δx is the displacement of the upper surface of the segment relative to the lower one, h is the thickness of the sample. For the specified steel segments after joint HPT at $\theta = 90°$ $\gamma_{real} = 5$. This is a large deformation, although less than $\gamma_1 = 8$ predicted by formula (1) for the point $R = 2.5$ mm, $h = 0.5$ mm. Thus, some slippage is observed already at the initial stage of HPT of mild steel.

Experiment IV. The sample, which is a disc of low-carbon steel Fe-0.1%C, is first subjected to HPT at $n = 5$ revolutions, then cut into segments and then these segments were subjected to joint HPT at $\theta = 90°$. The appearance of the segments indicates that the sample did not receive any noticeable shear deformation (Fig. 1f). Thus, after HPT $n \geq 5$, the surface of anvils and the surface of the sample slip and shear deformation does not occur.

During HPT, shear deformation occurs under the condition that the specific friction force F_μ between the surface of the anvils and the surface of the sample is greater than the yield stress (YS) of the material: $F_\mu > \text{YS}$. The specific friction force F_μ is determined by the equation $F_\mu = P \cdot \mu$, where P is the pressure, and μ is the friction coefficient. The pressure can be calculated as $P = U/S$, where U is the press force, and S is the area, and S is usually taken as the area of the anvils [3]. But in reality, a large area falls into the pressure zone, taking into account the edges of the working area of the anvils and the flash of the material, which reduces the specific pressure [3]. In our case, the press force is $U = 60$ t, the diameter of the working part of the anvils is $d = 1$ cm, so the area of the anvils is $S = 0.8$ cm^2, and for such an area the pressure will be $P = 6$ GPa. However, taking into account the flash, the sample-anvil contact area is $S \approx 1.5$ cm^2 and the actual pressure is much lower, about 3 GPa. The friction coefficient in HPT is estimated as $\mu = 0.3$ [14]. Hence $F_\mu \approx 1$ GPa, more than the YS of the original copper and low-carbon steel. But with an increase in the degree of deformation, metals are hardened, copper after HPT hardens to YS ≈ 1 GPa [2], which is less or comparable to F_μ. Low-carbon steel Fe-0.1%C after HPT is hardened to YS > 1.5 GPa [6], which exceeds F_μ. Hence, with an increase in the degree of HPT above a certain critical one, shear deformation of Fe-0.1%C is not implemented. The obtained result testifies in favor of the relevance of using accumulating HPT to achieve really large strains [15].

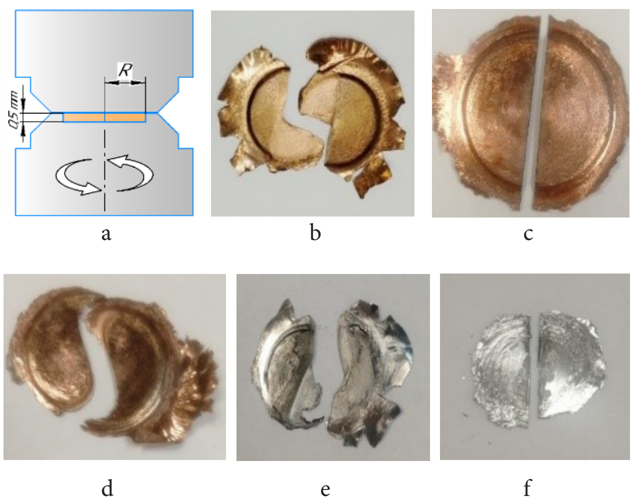

Fig. 1. (Color online) Scheme of joint HPT of halves to determine the degree of torsional deformation (a), copper halves after joint HPT $\theta = 90°$ (b), copper disk cut into segments after HPT $n = 5$ (c), same segments after subsequent joint HPT $\theta = 90°$ (d), Fe-0.1%C halves after joint HPT $\theta = 90°$ (e), Fe-0.1%C segments after HPT $n = 5$ + cutting + joint HPT $\theta = 90°$ (f).

The slippage effect must certainly depend on a number of parameters of HPT — pressure, design of anvils, their rotation speed, and others. It can be assumed that the slippage observed in our work is a special case and is due to the HPT parameters/setting used in this work. In this regard, it is of interest to study the microstructure of Fe-0.1%C samples subjected to HPT on our facility, and by the HPT regime usually used by other authors. Due to this, Fe-0.1%C solid discs were subjected to HPT for $n = 5$ and 10 revolutions at $P = 6$ GPa at room temperature on our HPT equipment

Fig. 2 shows the structure of the studied steel in TEM after HPT for $n = 5$ and 10 revolutions in a bright field (Fig. 2a, c) and a dark field (Fig. 2b, e) modes, as well as microdiffraction patterns in inserts. On the electron diffraction pattern of Fe-0.1%C after HPT for $n = 5$ and $n = 10$, several large reflections and many small ones blurred azimuthally and radially are observed along the ring (Fig. 2, insets). The general view of the electron diffraction patterns indicates a strong refinement of the structure.

In the bright and dark TEM fields, after HPT $n = 5$ and 10, a finely dispersed and inhomogeneous structure is observed. Fragments of the structure are stretched. It is difficult to determine the average grain size from bright-field images due to strong work hardening; however, the size of the structure fragments (band width) after HPT $n = 10$ is somewhat smaller than after HPT $n = 5$. According to dark field TEM, after HPT $n = 5$, the average grain/subgrain size is about 200 nm, after HPT $n = 10$ — about 150 nm, that is, the structure is additionally refined in comparison with the state of HPT $n = 5$. In general, the structure is similar to that observed in the works of other authors on same material [6].

According to XRD data, HPT of steel Fe-0.1%C results in decreased coherent scattering regions (CSR) and growing density of dislocation, while after HPT $n = 10$, the density of dislocation is much higher than after HPT $n = 5$. HPT results in a significant growth of HV (Table 1), as observed in [6, 7]. After HPT for $n = 10$ revolutions, HV is much higher than after HPT $n = 5$ (Table 1). Thus, the microhardness values of steel after HPT with the used mode and facility, in spite of the recorded slippage, reach approximately the same values as in

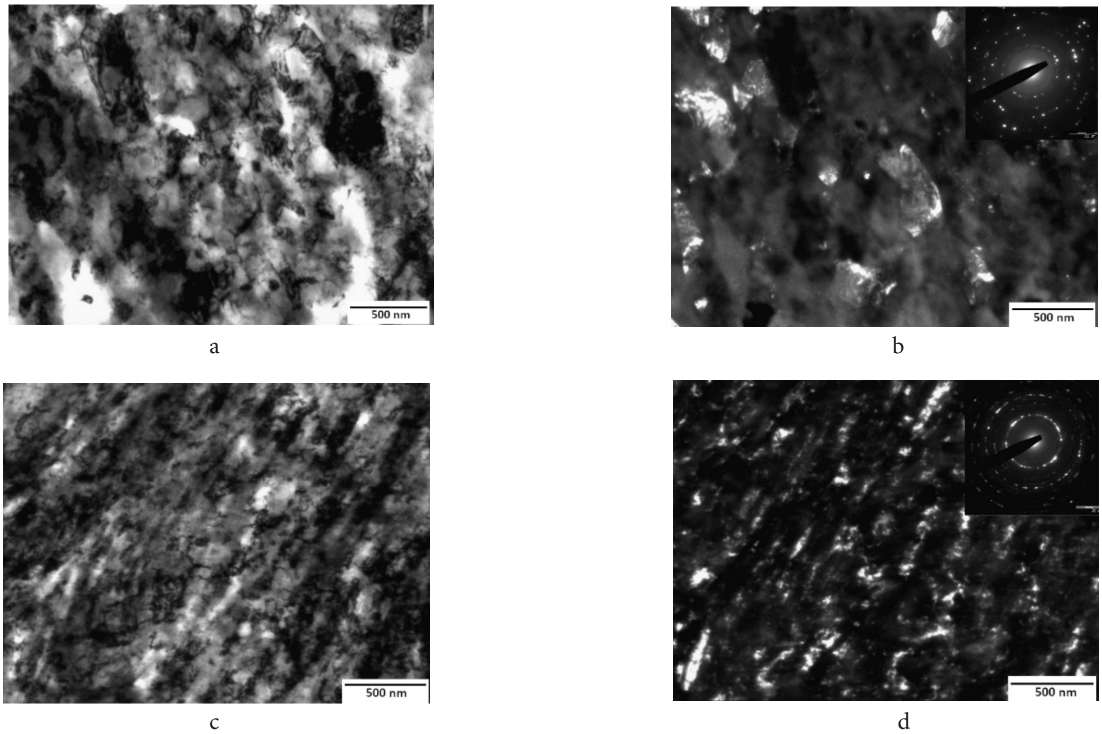

Fig. 2. TEM images of the structure of low-carbon steel Fe-0.1%C after HPT $n=5$, bright field (a) and dark field (b), diffraction pattern (inset); after HPT $n=10$, bright field (c), dark field (d), diffraction pattern (inset).

Table 1. XRD and hardness results before and after HPT.

Condition	D, nm	ε, %	ρ, 10^{15} m^{-2}	HV0.1 in the region ½ R
Initial	118	0.15	0.8	230
HPT $n=5$	25	0.35	2.8	400
HPT $n=10$	24	0.62	5.5	650

D is the size of coherent scattering regions: ε — grid distortions; ρ — density of dislocation.

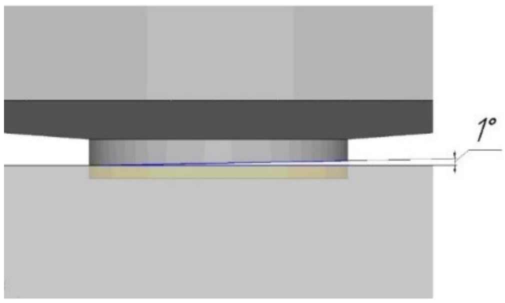

Fig. 3. HPT scheme in which the planes of the upper and lower anvils are declined from each other by 1°.

the works of other authors [6,7]. In addition, TEM, XRD and HV data unambiguously indicate that the structure after HPT $n=10$ is more refined and riveted than after HPT $n=5$. At the same time, experiment IV evaluating slip shows that there is no shear deformation after HPT $n>5$. Hence, despite the slip at HPT, when $n\geq 5$, deformation is still carried out, but not by relative torsion of the upper and lower parts of the sample, as is assumed by formula (1), but in some other way.

How can the accumulation of strain occur at HPT if slippage occurs at n more than 5? One of the possible explanations for the accumulation of strain in the sample during HPT, despite slippage, can be the following: it can be assumed that the planes of the upper and lower anvils are declined from each other by a small degree (for example, by 1°) (Fig. 3). With the length of the guide columns of the press about 2 m, the area of the press platforms 1 m^2, and the diameter of the working part of the anvil 10 mm, such a deviation is possible, and even inevitable. In this case, when the anvils rotate relative to each other, the sample material under a pressure equal to several GPa will flow from one deformation zone under the anvils to another. This will deform the material and modify its structure [16].

The sample subjected to such a deformation pattern was simulated using finite element computer modeling in the Deform-3D software package. In the simulation, a model of a workpiece with a circular cross-section with a diameter of 20 mm, and a groove on anvils with a depth of 1 mm was used. Steel 52100 from the Deform 3D library was selected as the workpiece material. At the first step of simulation, preliminary upsetting was carried out to uniformly fill the deformation zone and establish contact with the anvils over the entire area of the workpiece. At the second step, the torsion operation was simulated.

Workpiece and tool models were created in the KOMPAS-3D software package and imported in .stl format. The generated finite element mesh consisted of 32000 tetrahedra. The option to volume compensation of the workpiece was activated. The original workpiece was a plastic body, and the tool was an absolutely rigid body. Tool models were not meshed.

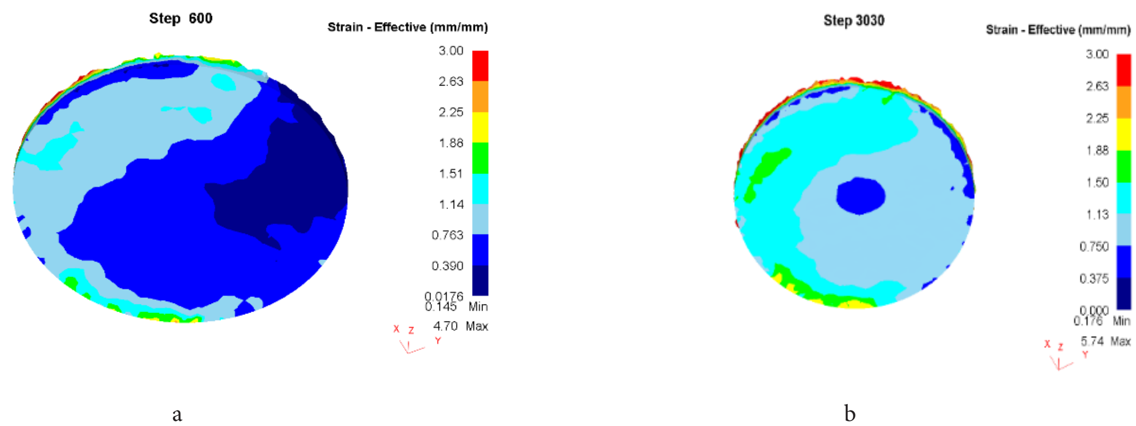

Fig. 4. (Color online) Simulation results: accumulated strain with an inclination of the anvils by 1° with full slip of the upper anvil over the surface of the sample at HPT $n=1$ (a), and HPT $n=5$ (b).

The rotation speed of the driven anvil was chosen constant and equal to 1 rpm. The workpiece temperature was taken constant and equal to room temperature (20°C). To set contact between the rotating anvil and the workpiece, the Siebel friction factor $f=0.12$ was used, bonded contact was used between the fixed anvil and the workpiece. The non-insight condition was set on the contact surfaces of the equipment. The number of modeling steps was over 1000.

Fig. 4 shows the results of modeling the accumulated strain with an inclination of the anvils by 1° with full slip of the upper anvil over the surface of the sample.

The simulation results show that, in spite of slippage, with such a scheme, the accumulated strain increases with an increase in the number of revolutions of the anvils. At $n=1$, the accumulated strain reaches the values $e\approx 0.7-1.14$, and at $n=5$ the strain reaches $e\approx 1.13-1.88$ (Fig. 4). It should be noted that $e\approx 2$ is a quite large strain, which, taking into account the high applied pressure, should lead to the formation of a nanostructure.

The issue of slippage during HPT of various materials, of the ways of accumulation of deformation during HPT is undoubtedly complex and multifactorial. For example, anvil roughness, which prevents slippage, may play an important role in HPT [12]. In the accumulation of deformation during HPT, the deformation by upsetting [1] can be of decisive importance, which increases with decreasing sample thickness with an increase in the number of revolutions of HPT. Also, according to the used method, we stopped the deformation after $n=5$, cut the sample into pieces, and returned to the anvils. But in this case, the geometric conformity between the sample and the tooling is lost. As a consequence, the contact area is expected to be much lower in interrrupted tests. These issues require further research and analysis.

4. Conclusions

It has been shown that slippage at the initial stages of high-pressure torsion deformation of copper and Fe-0.1%C steel is insignificant, while after HPT for $n=5$ revolutions, torsional deformation of steel Fe-0.1%C does not occur due to slippage.

TEM, XRD and HV data unambiguously indicate, that the structure after HPT $n=10$ is more refined and riveted than after HPT $n=5$. Hence, despite the slip at HPT, when $n\geq 5$, deformation is still carried out, but not by shear, as is assumed by formula (1), but in some other way. The structure of Fe-0.1%C formed after HPT in this research is similar to that observed in other papers [6,7], which allows suggesting that in most papers on HPT of Fe-0.1%C, slippage also occurs at some stage of straining, though the authors did not evaluate this.

One of the possible explanations for the accumulation of strain in the sample during HPT, despite the slippage, may be that the planes of the upper and lower anvils are declined from each other by a small angle. This leads to the accumulation of significant strain during the rotation of the anvils.

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References

1. A.P. Zhilyaev, T.G. Langdon. Prog. Mater. Sci. 53, 893 (2008). Crossref
2. R.Z. Valiev, A.P. Zhilyaev, T.G. Langdon. Bulk Nanostructured Materials. Hoboken, NJ, John Wiley & Sons, Inc (2013). Crossref
3. K. Edalati, Z. Horita. Mater. Sci. Eng. A. 652, 325 (2016). Crossref
4. R.Z. Valiev, Y.V. Ivanisenko, E.F. Rauch, B. Baudelet. Acta Mater. 44, 4705 (1996). Crossref
5. A.A. Popov, I.Y. Pyshmintsev, S.L. Demakov, A.G. Illarionov, T.C. Lowe, A.V. Sergeyeva, R.Z. Valiev. Scr. Mater. 37, 1089 (1997). Crossref

6. A. V. Ganeev, M. V. Karavaeva, X. Sauvage, E. Courtois-Manara, Y. Ivanisenko, R. Z. Valiev. IOP Conf. Ser. Mater. Sci. Eng. 63, 012128 (2014). Crossref
7. S. V. Dobatkin, P. D. Odessky, S. V. Shagalina. Mater. Sci. Forum. 584 – 586, 623 (2008). Crossref
8. P. W. Bridgman. Phys. Rev. 48, 825 (1935). Crossref
9. K. Edalati, Z. Horita, T. G. Langdon. Scr. Mater. 60, 9 (2009). Crossref
10. D. Kuhlmann-Wilsdorf, B. C. Cai, R. B. Nelson. J. Mater. Res. 6, 2547 (1991). Crossref
11. D. V. Gunderov, A. A. Churakova, E. V. Boltynjuk, E. V. Ubyivovk, V. V. Astanin, R. N. Asfandiyarov, R. Z. Valiev, W. Xioang, J. T. Wang. J. Alloys Compd. 800, 58 (2019). Crossref
12. Y. Huang, M. Kawasaki, A. Al-Zubaydi, T. G. Langdon. J. Mater. Sci. 49, 6517 (2014). Crossref
13. D. Gunderov, A. Stotskiy, Y. Lebedev, V. Mukaeva. Metals (Basel). 11, 573 (2021). Crossref
14. P. W. Bridgman. Proc. Am. Acad. Arts Sci. 71, 387 (1937). Crossref
15. D. V. Gunderov, D. A. Khasanova, V. V. Astanin, A. A. Churakova, I. A. Ramazanov, S. D. Prokoshkin, V. A. Sheremetyev. IOP Conf. Ser. Mater. Sci. Eng. 1008, 012013 (2020). Crossref
16. Patent RF № RU 159959 U1, 06.05.2015. (in Russian) [Патент RU 159959 U1, 06.05.2015]