Properties of Structural Steels with Nanoscale Substructure

T.V. Lomaeva, L.L. Lukin, L.N. Maslov, O.I. Shavrin and A.N. Skvortsov

Abstract To increase the reliability of products, the structural integrity of structural steels are relevant scientific challenges for materials specialists all over the world. A new direction of dealing with these challenges, i.e., making steels with superdispersed, including nanosized, structures, was formed during the past decade. Methods for obtaining such materials define their structural features (grain sizes, grain boundary interface development) and strength characteristics under different types of loading

Keywords Nanosizes · Structural steels · Nanopatterning · Processing methods Heating · Strain · Polygonization · Strength characteristics · Thermal strain processing

Introduction

Increasing the structural integrity of structural steel and engineering products due to nanotechnology in the world material science become a new scientific direction [5, p. 71; 9, p. 888; 3, p. 914].

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Nanopatterning strengthening effect of structural bulk metallic materials can increase the reliability and durability of engineering, energy-saving and source-saving products.

Researches carried out in this area [5, p. 71; 9, p. 888; 6, p. 546] allowed to develop methods of nanoscale structural materials production by dividing them into four groups:

- amorphous condition crystallization;
- powder metallurgy (nanopowder compacting);
- intensive plastic strain;
- various methods of nanocoating processes.

The authors think that grain size, morphology, and texture may change with respect to corresponding technological parameters of nanoscale material production process. Boundary interface volume fraction (grain boundaries and triple points) increases considerably with decreasing grain size, it has an essential influence on nanoscale material properties.

The criteria for present division into groups is only grain boundaries of materials and the effect they have on strength on structural materials. The works of Bernshtein M.L. school of thought [1, p. 211; 3, p.140] strongly indicate that the grain fine structure, formed by polygonal and cellular sub-boundaries, is the strength affecting factor even without significant grain size decrease.

Thermal Strain Processing

The size of fine structure elements, i.e., polygons and cells is determined by processing methods used for fine structure patterning. Types of thermomechanical processing from polygonal substructure patterning viewpoint can be united by a notion of thermal strain processing which becomes the fifth method of nanoscale structure patterning in structural materials.

The structure and operations used for this process, in general, may be similar to thermomechanical processing, especially for high-temperature thermomechanical processing (HTTMP) only in the set of operations, however, their parameters and some features are different.

As one can see from the literature, if the process of thermomechanical processing is performed at a conventional metallurgical production including steel making, crystallization, and multi-stage working of the finished section with quenching, then parameters of these operations being set by the desire to their optimization due to general production strategy, can provide only physical and mechanical characteristics improvement of metal.

Thermal strain process performed as a special strengthening processing the object of strengthening is a standard metal treated in accordance with the corresponding Standard GOST, i.e., a wire, hot-rolled, and calibrated steel, used as a workpiece for parts (e.g. spring wire), or some semi-finished product subjected to surface forming operations of a part, being subjected to strengthening thermal strain processing (TSP) provides improvement of its physical and mechanical characteristics due to nanoscale substructure patterning in the material of the part.

In case of thermomechanical processing, [4, p.76] performed during metallurgical production, the final operation in the processing chart is preceded by a number of technological conversions that are usually far from their optimal modes from the final result viewpoint—reaching the high-level strength characteristics.

In case of TSP, when the processing is performed as a special (separate) strengthening operation, one has to deal with metal subjected to all operations of metallurgical processing method. The TSP processing model, in this case, depends on part design, its end-use requirements and type of the workpiece used.

For a TSP object, like wire or calibrated steel, the processing model of thermal strain nanoscale substructure patterning includes basically the same list of operations. A workpiece for TSP operation to strengthen wire and calibrated steel can be:

1. hot-rolled wire or hot-rolled product;
2. cold-worked wire or calibrated steel.

Both workpiece types should be subjected to surface processing to remove the defect surface layer (decarburization, scaling removal, etc.). The quality of surface preparation is also determined by metal application area after strengthening processing.

The processing model of strengthening for wire and calibrated steel due to thermal strain nanoscale structure patterning includes the following operations:

1. fast heating of metal up to a temperature of homogeneous austenite formation (by its chemical composition), with full carbide solution.

Heating parameters (speed, temperature, holding) should provide austenite homogeneity without grain growth. The optimum is required to ensure homogeneity at the minimal size of initial austenite grain being further subjected to straining.

2. plastic metal strain. One should take into consideration the following parameters when choosing the value of strain:
   a. feasibility;
   b. loading straining model (SM) during operation of the produced part, which workpiece is being strained.
   c. the time of plastic straining.

The strain value may vary from 10–20% when using the plastic strain model taking into account SM of part operation.

Minimization of the time taken to plastic straining excluding development of annealing processes according to dynamic recrystallization model keeps the substructure nanoscale dimensionality.

The combination of three process parameters, i.e., heating temperature, strain value, and time and the time of strain holding are to provide steel polygonized substructure patterning prior to recrystallization. Under optimal combination, the substructure dispersity may correspond to nanoscale-dimensional range.

4. final operation is quenching.

The requirement to the final stage of nanoscale structure process is cooling, i.e., implementation of complete martensitic transformation.

Requirements to Parameters of Process Stages

I heating

1. temperature range 900–1000 °C;
2. speed V_{phase}°/sec 200–300.

The task is to provide disperse, homogeneous structure of chemical content.

Hence, the temperature should be minimal, while heating rate should be maximum. Primary chemical content homogenization should not contain unsolved secondary phase.

II straining

– Strain degree 10–25% by components of shear.

The task is to pattern a dislocation structure corresponding to hot–cold work dislocation structure [2, p. 20, 31] excluding recrystallization structure elements. To minimize strain value coincidence of straining models under strengthening high-temperature straining and operational loading.

III strain holding

– duration within 5–10 s.

The task is polygonal dislocation rearrangement to pattern a polygonized substructure of nanoscale dispersion.

IV cooling to quench the strained metal.

The cooling rate exceeds the critical quenching rate for the given steel.

Wire and Calibrated Steel TSP

In accordance with TSP concept, nanoscale structure patterning for structural steels can be performed on specialized equipment.

To perform TSP methods, a typical processing method variable for wire and calibrated steel strengthening. The processing method includes the following operations: initial material surface preparation, induction heating for straining, straining, strain holding, cooling (quenching) in continuous and sequential mode, and tempering.

Hot straining is performed as follows: for wire by drawing, for calibrated steel by spring setting in rotating head [8, p. 58; 7, p. 35] (Figs. 1 and 2).

The design scheme for calibrated steel with TSP production is shown in Fig. 1, it consists of a workpiece feeding device 2 to feed the workpiece 1, high-frequency heating device 3 and spring setting (SS) straining device 4, cooling sprayer 5 and a pulling mechanism 6. The deforming head consists of 3 rollers located at an angle of 120°, the head rotates at 500–700 rpm. The workpiece heating temperature before straining (900–1000 °C) and the diameter setting degree (15–25%). The workpiece being stretched after straining is cooled with water in the sprayer within controlled time to guarantee its straightness ($\Delta \leq 0.2$ mm/m) and exclude further restriking.

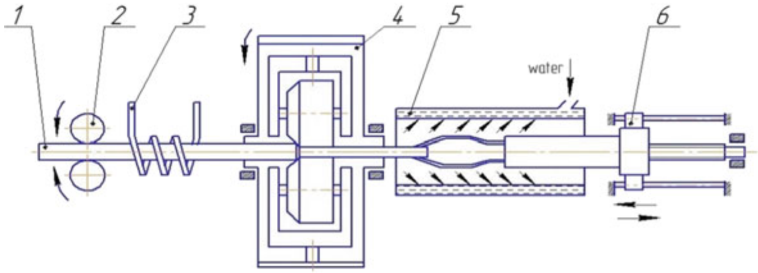

Fig. 1 Design scheme of TSP calibrated steel *1* workpiece, *2* feeding rollers, *3* induction block, *4* deforming head, *5* sprayer, *6* axial motion drive

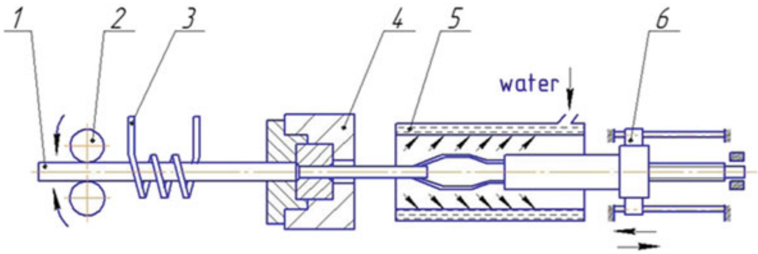

Fig. 2 Design scheme of TSP wire *1* workpiece, *2* feeding rollers, *3* induction block, *4* deforming drawing block, *5* sprayer, *6* axial motion drive

The design scheme for making TSP wire (Fig. 2) consists of the same functional assemblies as shown in Fig. 1., however, wire straining with strain degree up to 20% is performed in the hard-alloy die.

Wires made of 51XΦA (C 0.47–0.55%, Si 0.15–0.3%, Mn 0.3–0.6%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr 0.75–1.1%, V 0.15–0.25%, Cu up to 0.2%) and 60C2A (Fe ~ 96%, C 0.58–0.63%, Si 1.6–2%, Mn 0.6–0.9%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr up to 0.3%, Cu up to 0.2%) steel grades with finishing diameter 5 mm, and calibrated steel 9X (C 0.9%, Cr 1%) with diameter 16 mm were subjected to TSP.

Strength and plasticity characteristics were determined in accordance with Standard GOST 10446-80. Strength characteristics of steel 9X (C 0.9%, Cr 1%) for low-temperature tempering state were determined under concentrated load bending test due to low plasticity.

Fine structure of steels was studied on foils by means of electronic microscope EM-125 M under accelerating voltage of 100 kV.

Substructure images were computer processed, the crosswise size was measured—the shortest distance between elongated subgrain boundaries—elements of α-phase and carbides [8, p. 120]. The obtained results were statistically processed with the determination of the mean crosswise size of substructural element and root mean square deviation.

Electron microscopical studies showed that tested steels exhibited generally polygonal substructure patterning under TSP of the developed model—induction heating up to 900–1000 °C, strain degree 10–20% and rigidly controlled cooling, as shown in Fig. 3.

The study of wire heating temperature (920–1000 °C) made of steel 51XΦA (C 0.47–0.55%, Si 0.15–0.3%, Mn 0.3–0.6%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr 0.75–1.1%, V 0.15–0.25%, Cu up to 0.2%) at TSP (Fig. 3a–c, e) showed that wire structure is of principally same type, i.e., polygonal structure of parallel elements fragmented by equiaxial cells.

The average polygon crosswise size for wire heated before straining up to 920 °C is 51.9 nm, and wire heated up to 1000 °C it is 80.5 nm.

The principal structural difference of wires heated up to 920 and 1000 °C is in the occurrence of carbides and their sizes. Carbide sizes in wires heated up to 920 °C are bigger and reach 150 nm, the number of carbides in wires heated up to 1000 °C is considerably less and their size is also less. This goes to prove that temperature of 920 °C is not enough for carbide solution. It is necessary to increase the time of wire under high temperature before straining during TSP.

The study of strain degree effect at TSP of wire made of steel 60C2A (Fe ~ 96%, C 0.58–0.63%, Si 1.6–2%, Mn 0.6–0.9%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr up to 0.3%, Cu up to 0.2%) (Fig. 3e) it was found that nanoscale substructure being patterned even under strain degree 10% do not principally change under strain degree of 20%.

The structure is both polygonal with parallel polygons and cellular. Substructure element sizes correspond to nanoscale dimensionally criterion, i.e., average size is 70 nm.

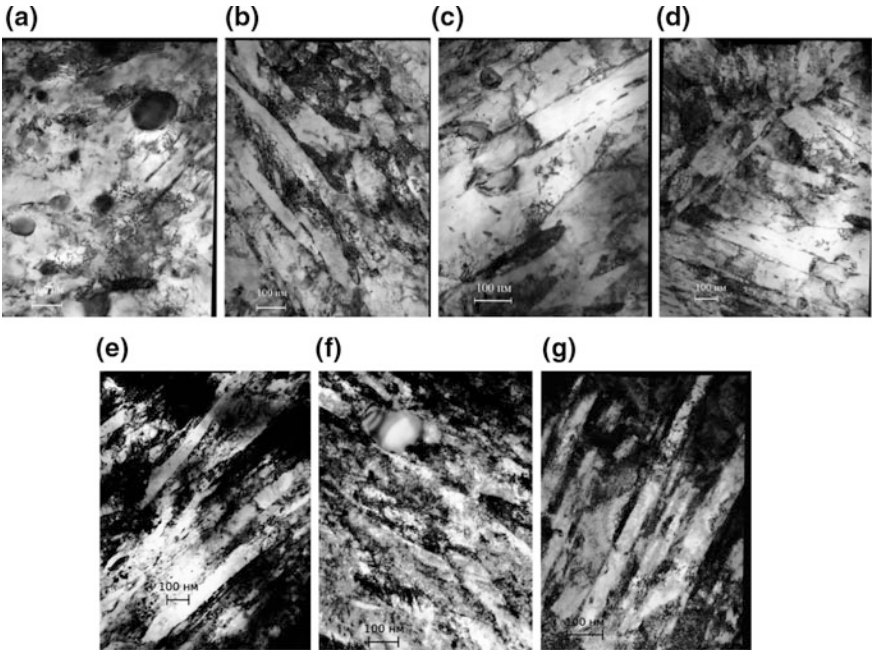

Fig. 3 Fine structure of steels after TSP. **a**, **b** wire made of steel 51XΦA (C 0.47–0.55%, Si 0.15–0.3%, Mn 0.3–0.6%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr 0.75–1.1%, V 0.15–0.25%, Cu up to 0.2%) T_{strain} = 920 °C; **c**, **d** wire made of steel 51XΦA (C 0.47–0.55%, Si 0.15–0.3%, Mn 0.3–0.6%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr 0.75–1.1%, V 0.15–0.25%, Cu up to 0.2%) T_{strain} = 1000 °C; **e** wire made of steel 60C2A (Fe ~ 96%, C 0.58–0.63%, Si 1.6–2%, Mn 0.6–0.9%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr up to 0.3%, Cu up to 0.2%) λ = 10%; **f** steel 9X (C 0.9%, Cr 1%); **g** steel 38XC (Fe ~ 95%, C 0.34–0.42%, Si 1.0–1.4%, Mn 0.3–0.6%, Ni up to 0.3%, S up to 0.035%, P up to 0.035%, Cr 1.3–1.6%, Cu up to 0.3%)

Ring electron diffraction pattern defines elementary α-phase cells with dominating orientation 110 with zone axis [001]. Azimuthal component of subgrain disorientation in this direction is 0.077 …. 0.096 rad.

The study of structural steels structure under TSP [38XC (Fe ~ 95%, C 0.34–0.42%, Si 1.0–1.4%, Mn 0.3–0.6%, Ni up to 0.3%, S up to 0.035%, P up to 0.035%, Cr 1.3–1.6%, Cu up to 0.3%) and 9X (C 0.9%, Cr 1%)] shown in Fig. 3f, g exhibited nanoscale substructure patterning. Steel grade affects the type of substructure elements either polygonal or cellular. Steel grade 9X (C 0.9%, Cr 1%) after TSP showed the occurrence of unsolved carbides under induction heating.

Test results of specimens made of steel 60C2A (Fe ~ 96%, C 0.58–0.63%, Si 1.6–2%, Mn 0.6–0.9%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr up to 0.3%, Cu up to 0.2%) 5 mm in diameter are given in Table 1. The effect of strain degree and tempering temperatures was studied. The check specimens were treated at conventional modes for steel 60C2A (Fe ~ 96%, C 0.58–0.63%, Si 1.6–2%, Mn 0.6–0.9%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr up to 0.3%, Cu up to 0.2%), i.e., furnace heating temperature constitutes 860 °C and oil cooling. Both check specimens and TSP specimens were tempered at temperature values of 200, 300, 400, and 460 °C.

The check specimens after low-temperature tempering at 200 °C show brittle fracture without plasticity features (absence of necking on fractured specimens). Plasticity features appear only after tempering at temperature 300 °C, i.e., $\delta = 3\%$, $\psi = 23\%$. Values of both δ and ψ correspond to the specified requirements and are observed after tempering at 460 °C.

The study of strain degree effect showed that for the applied TSP processing model the essential improvement of strength characteristics is observed at strain degree of 10%. After tempering at temperature value of 300 °C under the required plasticity ($\delta = 6.5\%$, $\psi = 36\%$) values of s_u, $s_{0.2}$, s_{pr} are 2480, 2070, 1990 MPa. Increasing strain degree up to 20% at slightly low values of δ (4.7%) allow obtaining s_u, $s_{0.2}$, s_{pr} values of 2500, 2290, and 1980 MPa correspondingly. The increased results were obtained for other tempering temperatures. This is the result of induction heating that allows obtaining relative combinations of structures and strength characteristics during industrial technology elaboration by varying the number of parameters.

Studying the TSP methods for calibrated steel 9X (C 0.9%, Cr 1%) the effect of strain temperature, strain degree, and tempering temperatures after quenching was examined.

The check specimens were taken after conventional heat treatment used for 9X (C 0.9%, Cr 1%) steel grade. The tempering temperatures were assigned by the necessity to study steel properties at high hardness stipulated by operational conditions.

Testing metals of high hardness under tension has two obstacles: difficulty in specimen manufacturing and noninformative results due to brittle fracture.

Taking all these facts into account strength characteristics were determined at bending test loaded by a concentrated load. Bending moment *M*, ultimate bending strength $s_{u\ bending}$ and deflection *f* mm were measured.

The results of specimen bending tests are given in Table 2.

As it follows from the results given above, strength characteristics are essentially improved after TSP, both strength—M, $s_{u\ bending}$ and plasticity—*f*. Maximum strength increase is obtained under 10% straining at straining temperature value of 900 °C after tempering at 150 °C, i.e., characteristics of M, $s_{u\ bending}$ and *f* are 3270 Nm, 3930 MPa and 18.2 mm correspondingly. Check specimens after the same tempering conditions showed the values of 1860 Nm, 2150 MPa and 4.4 mm correspondingly.

When raising straining temperature up to 1000 °C strength characteristic values decrease up to M, $s_{u\ bending}$ and *f* to 2910 Nm, 3430 MPa and 10.0 mm correspondingly.

The effect of tempering temperatures within the studied interval showed that increasing tempering temperatures leads to increasing of all values—M, $s_{u\ bending}$ and *f*—after tempering at 180 °C they reach values of 4190 Nm, 5230 MPa and

Table 1 Properties of wire specimens made of steel 60C2A (Fe ~ 96%, C 0.58–0.63%, Si 1.6–2%, Mn 0.6–0.9%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr up to 0.3%, Cu up to 0.2%), d = 5 mm

Processing mode	$T_{tempering}$, °C	s_u (MPa)	$s_{0.2}$ (MPa)	s_{pr} (MPa)	d (%)	y (%)	$(s_{u(TSP)} - s_{u(CHT)})/s_{u(CHT)}$ (%)	$(s_{0.2(TSP)} - s_{0.2(CHT)})/s_{0.2(CHT)}$ (%)	$(s_{pr(TSP)} - s_{pr(CHT)})/s_{pr(CHT)}$ (%)
Conventional heat treatment (CHT), $T_{heating}$ = 860 °C furnace, oil cooling	220	brittle fracture							
	300	2100	1970	1910	3	23			
	400	2000	1900	1800	4.5	40			
	460	1620	1520	1400	7.6	41			
TSP, 10%, T_{strain} = 1000 °C	220	2520	2120	2000	4.8	32			
	300	2480	2070	1990	6.5	36	18	5	4
	400	2280	2130	2010	9.2	42	14	12	12
	460	1650	1550	1420	10.1	40.1	2	2	1
TSP, 20%, T_{strain} = 1000 °C	220	2610	2330	2210	4.9	32			
	300	2500	2290	1980	4.7	37.5	19	16	4
	400	2280	2040	1960	5.7	45.1	14	7	9
	460	1640	1580	1440	10.7	42.6	1	4	3

Table 2 Properties of specimens made of calibrated steel 9X (C 0.9%, Cr 1%) (bending test)

Processing mode	$T_{tempering}$ (°C)	M (Nm)	$s_{u\ bending}$ (MPa)	F (mm)	HRC	($s_{u\ bending\ (TSP)} - s_{u\ bending\ (CHT)}$)/$s_{u\ bending\ (CHT)}$(%)	($M_{(TSP)} - M_{(CHT)}$)/$M_{(CHT)}$(%)	($F_{(TSP)} - F_{(CHT)}$)/$F_{(CHT)}$(%)
CHT, $T_{heating}$ = 800 °C oil cooling	100	1650	1970	3.0	64.9			
	150	1800	2150	4.4	64.7			
	180	2110	2510	7.6	62.2			
TSP, 10%, T_{strain} = 900 °C	150	3270	3930	18.2	68.1	83	81	313
TSP, 10%, T_{strain} = 950 °C	150	3040	3710	13.7	66.0	72	69	211
TSP, 10%, T_{strain} = 1000 °C	150	2910	3430	10.0	66.0	59	62	127

18.5 mm. Straining was performed at a temperature of 950 °C, the strain degree constituted 20%. However, increasing tempering temperatures reduced the HRC hardness by 4 units.

Conclusions

1. In the result of TSP according to the elaborated processing model, it was found that nanoscale substructure patterning in spring wire made of steel 51ХФА (C 0.47–0.55%, Si 0.15–0.3%, Mn 0.3–0.6%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr 0.75–1.1%, V 0.15–0.25%, Cu up to 0.2%) and steel 60C2A (Fe $\sim$ 96%, C 0.58–0.63%, Si 1.6–2%, Mn 0.6–0.9%, Ni up to 0.25%, S up to 0.025%, P up to 0.025%, Cr up to 0.3%, Cu up to 0.2%) and for structural steel 9X (C 0.9%, Cr 1%).
2. The TSP method used for nanoscale substructure patterning provides an improvement of strength and plastic characteristics of spring and structural steels.
3. Maximum strengthening effect is observed when comparing the strength of steels subjected to TSP and to conventional heat treatment under the same plasticity level.

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