POSSIBILITY OF GRAIN REFINEMENT OF LOW CARBON STEEL BY CYCLING OF TEMPERATURE OR DEFORMATION

KAWULOK Petr[1], BULAWA Marek[1], FOLTA Ondřej[1], KAWULOK Rostislav[1], SCHINDLER Ivo[1], RUSZ Stanislav[1], OPĚLA Petr[1], SUBÍKOVÁ Miroslava[1]

[1]VSB - Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, 17 listopadu 15, 708 33 Ostrava - Poruba, Czech Republic, EU, petr.kawulok@vsb.cz, marek.bulawa.st@vsb.cz, ondrej.folta.st@vsb.cz, rostislav.kawulok@vsb.cz, ivo.schindler@vsb.cz, stanislav.rusz2@vsb.cz, petr.opela@vsb.cz, miroslava.subikova@vsb.cz

Abstract

Universal plastometer Gleeble 3800, which is installed at the Regional materials science and technology centre at the VSB - Technical University of Ostrava was used for investigation of possibility of grain refinement of low-carbon steel. Through dilatometric tests of the investigated steel performed at various heating and cooling rates, the transformation temperatures Ar_1, Ar_3, Ac_1 and Ac_3 were determined. These temperatures were employed at the proposal of two following types of experiments, performed to refine a prime grain of the examined steel. The first set of the tests contained an annealing off the examined steel with a cyclical change of the temperature. It was found by metallographic analysis of the annealed samples that the samples annealed at the temperatures of 880 and 530 °C with the dwell of one second at the annealing temperature, and cooled down or heated up at the rate of 10 °C·s^{-1} showed finer grain even after one cycle. Increase of the number of cycles caused a decrease of ferritic grain. The second set of the test contained a cyclic deformation of the examined steel, which consisted of combination of pressure and tension at the given temperature. The deformation temperature, the size of the absolute strain, the dwell time between the individual cycles and the number of the individual cycles were chosen as variable parameters. Metallographic analysis of each cyclically deformed sample proved that the selected parameters of deformation did not lead to the required refinement of microstructure of the examined steel.

Keywords:

Grain refinement, temperature of phase transformation, cycling of temperature, cycling of deformation, plastometer Gleeble 3800.

1. INTRODUCTION

Obtaining of suitable material microstructure makes it possible to obtain higher strength properties and optimum fatigue and brittle-fracture characteristics. Homogeneous fine grain microstructures can be achieved by annealing of the material after its conventional forming or by its controlled forming [1-3]. In both cases, in addition to deformation and cooling parameters, the course of restoration processes and phase transformations has a significant effect on grain refinement [4-7]. The influence of thermo-mechanical; conditions on evolution of microstructure during forming can be investigated in laboratory by plastometric methods, which are based on pressure, torsion or cyclic deformation of the tested sample [8-10].

The aim of the experimental work was to explore the possibility of grain refining in structural low-carbon steel S355J2 by the temperature or deformation cycling. Plastometer Gleeble 3800, which is installed at the Regional materials science and technology centre at the VSB-TU Ostrava was used for all experimental works [8]. Its advantage consists in the possibility of realisation of various different thermo-mechanical tests on one device [8, 11, 12].

2. EXPERIMENT DESCRIPTION

Experimental works were divided into several parts. First of all it was necessary to determine temperatures of phase transformations of the investigated steel at its heating and cooling, which were then used for setting of temperature conditions of tests with temperature or deformation cycling. Structural low-carbon steel S355J2 with chemical composition (in mass %) 0.184 % C; 1.34 % Mn; 0.231 % Si; 0.015 % P; 0.005 % S; 0.04 % Cu; 0.05 % Cr; 0.02 % Ni; 0.029 % Al; 0.007 % Mo; 0.01 % W; 0.002 % V; 0.015 % Ti; 0.002 % Nb; 0.0048 % N was used for these purposes.

2.1 Determination of temperatures of phase transformations

Temperatures of phase transformations at heating (Ac_1, Ac_3) and cooling (Ar_1, Ar_3) were determined on the basis of evaluation of dilatometric tests. For this purpose cylindrical samples with diameter of 10 mm and length of 86 mm were prepared from the examined steel. The prepared samples were heated by electrical resistance heating at the rate of 1 °C·s^{-1} or 10 °C·s^{-1} to the temperature of 950 °C, followed by a 10 second dwell time at this temperature. The tested samples were then cooled down again at the rate of 1 °C·s^{-1} or 10 °C·s^{-1} to the temperature of 25 °C. During testing the temperature on the sample surface was measured by thermocouple wires. Sample dilatation during testing was measured by contact LVDT dilatometer with the range of ± 2.5 mm and resolution of ± 0.4 μm.

For the heating or cooling rate of 1 °C·s^{-1} the following temperatures of phase transformations Ac_1 = 722 °C, Ac_3 = 830 °C, Ar_1 = 609 °C, Ar_3 = 753 °C were determined. In the case of test at the heating or cooling rate of 10 °C·s^{-1} the following temperatures of phase transformations Ac_1 = 721 °C, Ac_3 = 850 °C, Ar_1 = 551 °C, Ar_3 = 720 °C were determined.

2.2 Tests with cyclic change of temperature

The samples with diameter of 10 mm and length of 86 mm were also used for plastometric tests with cyclic change of temperature, which in principle consisted of material annealing with temperature cycling. Individual annealing temperatures for these tests were determined on the basis of evaluation of dilatometric tests in such a manner, that phase transformation takes place in the examined steel at the temperature cycling.

The samples Z1 (1 cycle), Z2 (3 cycles) and Z3 (10 cycles) were heated by electric resistance heating at the heating rate of 10 °C·s^{-1} to the lower annealing temperature of 530 °C. They were then heated at the heating rate of 10 °C·s^{-1} to the upper annealing temperature of 880 °C, and after 1 second dwell time at this temperature they were cooled down at the cooling rate of 10 °C·s^{-1} to the lower annealing temperature of 530 °C. After achievement of this temperature, in the case of multi-cycle test, again 1 second dwell time was held at this temperature and it was followed by the next cyclic heating and cooling. After completion of temperature cycling all the samples were cooled down from the lower annealing temperature at the cooling rate of 10 °C·s^{-1} to the temperature of 25 °C. In case of the samples Z4 (1 cycle), Z5 (3 cycles) and Z6 (6 cycles) were selected the lower annealing temperature of 500 °C, the upper annealing temperature of 860 °C, 15 second dwell time at those temperatures, and heating or cooling rate between those temperatures of 1 °C·s^{-1}. The remaining parameters were analogical to those for testing of the samples Z1 to Z3. All cyclically annealed samples were then subjected to metallographic analysis.

2.3 Tests with deformation cycling

Samples with diameter of 12 mm and overall length of 120 mm were prepared from the examined steel for tests with cyclic strain. This type of samples, with the diameter of the deformed part of the sample of 10 mm and length of 12 mm, is primarily determined for accelerated creep tests, which also represent deformation cycling during the test [13].

In order to ensure that deformation takes place in a single phase austenitic area, the deformation temperature of 880 °C was determined on the basis of evaluation of dilatometric tests. The prepared samples were heated by electric resistance heating at the heating rate of 10 °C·s^{-1} to the specified deformation temperature, followed by 60 second dwell time at this temperature. The samples were then cyclically deformed by alternating compression and tension at constant speed of travel of the crosspiece of 0.1 mm·s^{-1}, to which the strain rate of 0.01 s^{-1} corresponds, and at constant temperature with dwell times between reductions of various duration. One cycle represented deformation by alternating compression and tension, dwell time of temperature and deformation by alternating compression and tension. The samples were after deformation cooled down at the cooling rate of 2 °C·s^{-1} to the temperature of 500 °C, and then at the cooling rate of 10 °C·s^{-1} to the temperature of 25 °C.

The variable parameters were the magnitudes of engineering strain, which was in case of the samples D1 to D3 equal to 10 %, or in case of the samples D4 to D6 to 15 %, to which the magnitude of absolute length compression or tension strain of 1.2 mm or 1.8 mm corresponded respectively. Duration of the pause between reductions for the samples D1 to D3 was 10 seconds, in case of the samples D4 to D6 this pause between reductions was shortened to 7 seconds. The samples D1 and D4 were subjected to a 1-cycle deformation, the samples D2 and D5 were subjected to a 3-cycle deformation, and the samples D3 and D6 were subjected to a 10-cycle deformation. All deformed samples were subjected to metallographic analyses.

3. PROCESSING OF MEASURED DATA AND DISCUSSION OF RESULTS

During tests with cyclic change of temperature apart from the temperature also the sample dilatation was registered. Example of comparison of dilatation of the samples at the tests with temperature cycling is documented in **Fig. 1**. Already these dilatation curves suggested that during these tests the grain was refined with the increasing number of cycles, which was manifested by reduction of differences between Ar_3 temperatures between individual cycles.

Fig. 2, illustrating drop of force during the test with 10-cycle deformation (sample D6), indicates that during these tests the required grain refinement did not take place, since the so called cyclic softening occurred during this test [14].

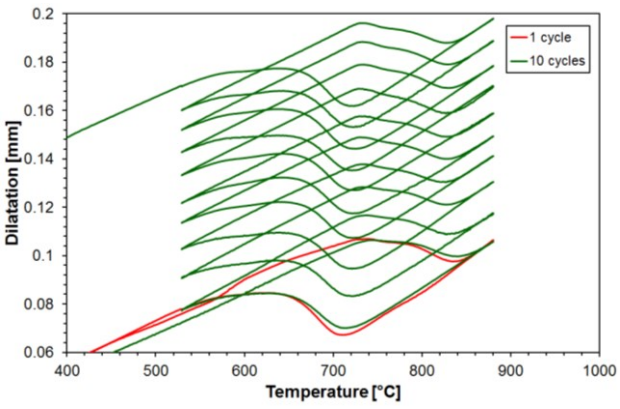


Fig. 1 Example of dilatation of samples during tests with cycling change of temperature

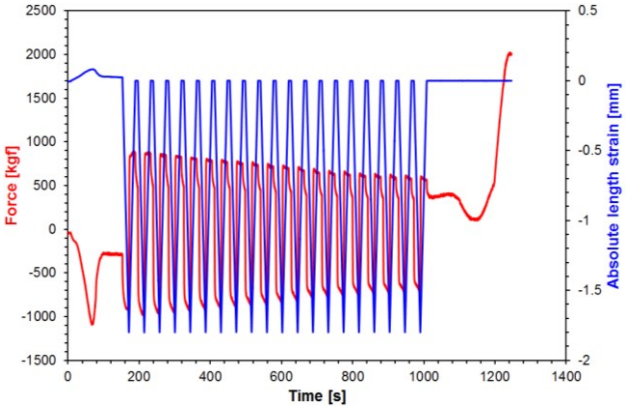


Fig. 2 Evolution of force and strain in dependence on time at the test with 10-cycle strain (sample D6)

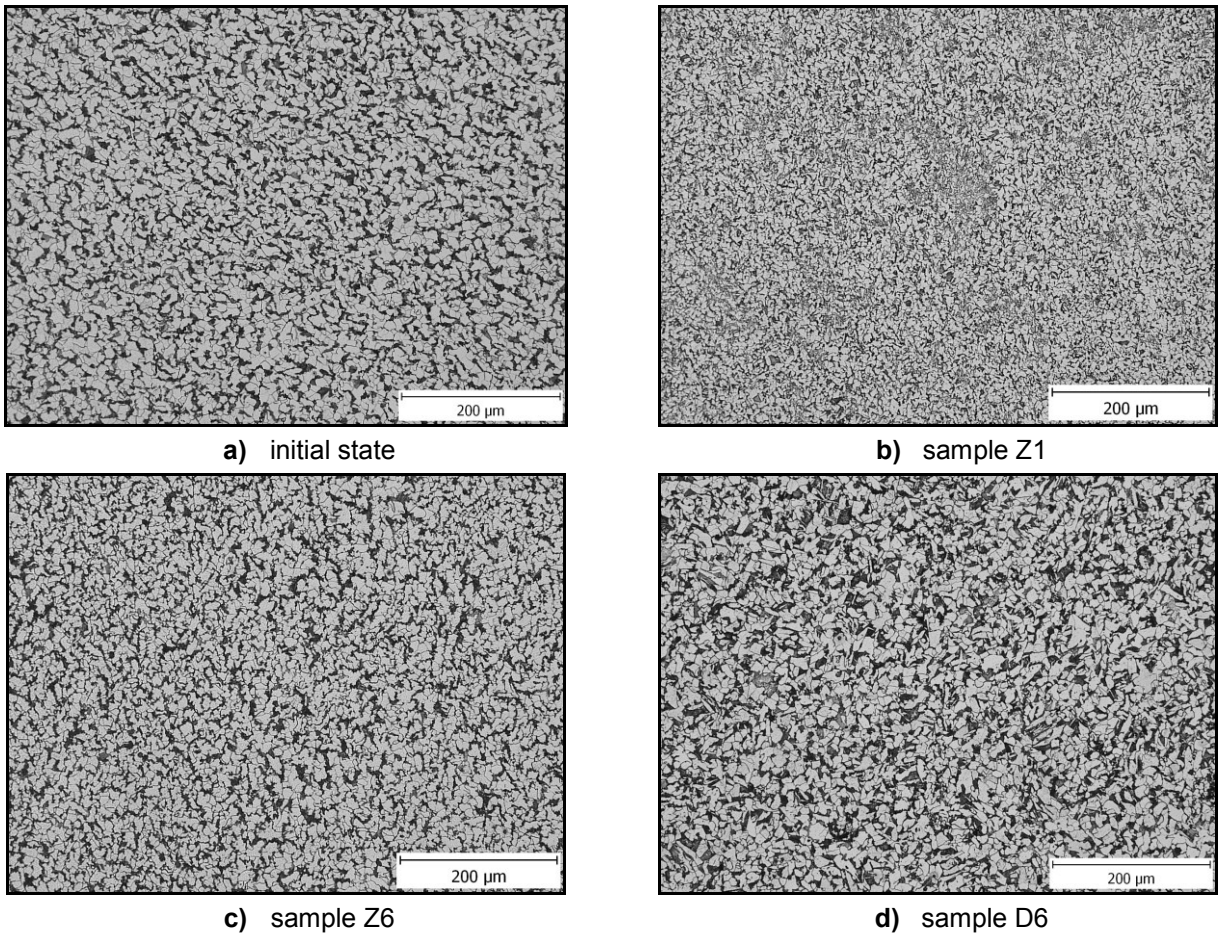
The initial homogeneous micro-structure of the examined low-carbon steel was formed by a mixture of ferrite and pearlite, while the mean size of ferritic grain, determined by linear method, was 11 μm – see **Fig. 3a**. Already at single-cycle change of temperature at annealing with comparatively high heating and cooling rate of 10 °C·s^{-1} (sample Z1) relatively considerable refinement of ferritic grain was achieved – see **Table 1** and **Fig. 3b**, while with the increasing number of cycles the grain refinement was more significant. In the case of

cyclically annealed samples heated and cooled at the rate of 1 °C·s^{-1} such significant grain refinement did not take place – see **Fig. 3c**.

Metallographic analysis of cyclically deformed sample confirmed that in case of these samples the required refinement of ferritic grain did not take place see - **Table 1** and **Fig. 3d**. With the increasing number of cycles the ferritic grain got even slightly coarser, which contradicts the findings presented by the authors [15], who claim that repeated deformation does nod enable growth of recrystallized grains. The cause of grain coarsening might have been in this case a combination the austenitisation itself of the samples and deformation by alternating compression and tension, which probably limited the effects of static recrystallization.

Table 1 Mean grain size of the tested samples

Annealed sample	**Z1**	**Z2**	**Z3**	**Z4**	**Z5**	**Z6**
Mean grain size [μm]	7.3	6.2	5.5	10.8	10.6	10.3
Deformed sample	**D1**	**D2**	**D3**	**D4**	**D5**	**D6**
Mean grain size [μm]	11.8	12.0	13.3	11.6	12.2	12.6



a) initial state **b)** sample Z1

c) sample Z6 **d)** sample D6

Fig. 3 Microstructure of tested samples

In order to suppress the influence of duration of pause between compression or tension deformation, another two tests were made with 10-cycle strain without pauses between reductions, the aim of which was to initiate in material dynamic recrystallization. Magnitude of overall logarithmic length strain (true strain) in the case of 10 cycling tests was equal to 6.5. The first test was performed at analogical deformation temperature as in previous tests = 880 °C. For the second test the deformation temperature 930 °C was chosen.

Metallographic analysis of thus deformed samples was documented, that elimination of inter-pass time and expected course of dynamic recrystallization did not have a positive effect on the final size of the ferrite grain. The resulting micro-structure of thus deformed samples was again formed by a mixture of ferrite and pearlite, while size of ferritic grain was 13.4 μm in the case of deformation temperature of 880 °C, or 13.7 μm in the case of deformation temperature of 930 °C.

For verification conditions for course of dynamic recrystallization another two continuous tests were moreover performed by uniaxial compression at the deformation temperatures of 880 and 930 °C and the strain rate of 0.01 s^{-1} with logarithmic length strain of -1.5. For these purposes were prepared from investigated steel samples with diameter of 10 mm and height of 15 mm. Parameters of heating and cooling were analogical to those used in the case of cyclically deformed samples. It is evident already from the recorded stress-strain curves (see **Fig. 4**) that dynamic recrystallization was initiated in material in the course of those tests. Microstructure of the samples after continuous test by uniaxial compression was formed by a mixture of equiaxed ferritic grains and slightly stretched pearlitic formations – see **Fig. 5**. Average size of the resulting ferirtic grain was at the deformation temperature of 880 °C equal to 9.9 μm, or to 10.7 μm in case of the sample deformed at the temperature of 930 °C. The samples deformed by uniaxial compression showed slight grain refinement in comparison with cyclically deformed samples, the real length deformation of which was approx. 4 times bigger.

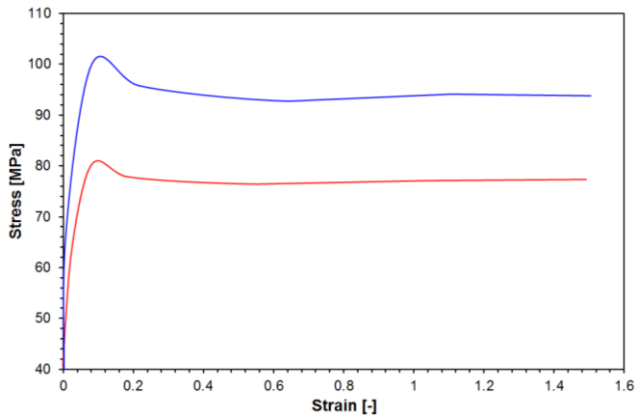


Fig. 4 Stress-strain curves obtained by continuous tests by uniaxial compression

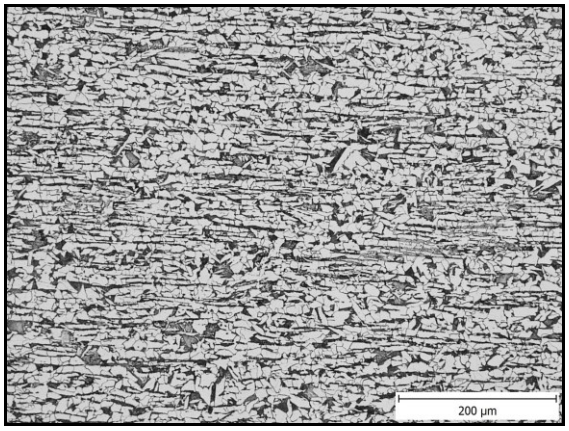


Fig. 5 Microstructure of the sample after continuous compression test at the temperature of 880 °C

4. CONCLUSIONS

Using the physical simulations performed on the plastometer Gleeble 3800 the possibilities of grain refinement of low-carbon steel S355J2 by temperature or strain cycling were investigated.

Use of the evolution of phase transformations initiated by temperature cycling of the tested samples made it possible to achieve the required refinement of resulting ferritic grain in the examined steel, while the size of ferritic grain was getting refined with the increasing number of cycles. Temperature cycling enabled achievement of homogeneous microstructure formed by a mixture of pearlite and ferrite with size even 5.5 μm.

In the case of tests with cyclic deformation of the samples the required grain refinement was not achieved. With the increasing number of cycles, consisting of alternating effect of compression and tension on the tested sample, the size of the resulting ferritic grain even slightly increased. On the other hand comparison continuous tests by uniaxial compression, performed under analogical temperatures and strain rate as the tests with cyclic deformation, but at approx. 4 times smaller logarithmic length strain, initiated dynamic recrystallization and slight refinement of ferritic grain.

Very important finding consists in the fact that material deformation based on cyclic changes of tension and compression (see e.g. [10, 16]) need not result in identical structural characteristic as after analogical deformation, performed by compression only. From this perspective the simulation of forming processes on classical compression or (in the case of big accumulated strains) on torsion plastometers seems to better reflect the real industrial operation conditions [8].

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