2009

J. KRAWCZYK*, P. BAŁA*

OPTIMALIZATION OF HEAT AND THERMO-CHEMICAL TREATMENT OF 50CrMoV18-30-6 STEEL FOR HOT FORGING DIES

OPTYMALIZACJA OBRÓBKI CIEPLNEJ I CIEPLNOCHEMICZNEJ STALI 50CrMoV18-30-6 NA MATRYCE DO KUCIA NA GORĄCO

This work discusses an effect of technology of heat and thermo-chemical treatment on properties of the 50CrMoV18-30-6 steel for hot forging dies. The quality of the microstructure of the investigated steel was evaluated in the as-delivered condition (as-delivered to forging dies producers). Basing on the series of hardening heat treatments and dilatometric tests (heating curves), the optimal austenitizing temperature for heat treatment of the steel was determined. The CCT diagram and the simulation of complex heat treatment was performed on DT 1000 dilatometer. The reasons for the occurrence of failures in dies made of that steel were discussed. Within a framework of this research, dislocation structure of a damaged die as well as the microstructure of the nitrided layer was discussed.

W pracy przedstawiono problem wpływu technologii obróbki cieplnej i cieplno-chemicznej na własności użytkowe matrycy do kucia na gorąco wykonanej ze stali 50CrMoV18-30-6. Określono jakość mikrostruktury badanej stali w stanie dostarczanym do odbiorców (producentów matryc). Na podstawie "szeregu hartowniczego" oraz pomiarów dylatometrycznych (krzywej nagrzewania) określono właściwą temperaturę austenityzowania do obróbki cieplnej tej stali. Wykonano symulację całego procesu obróbki cieplnej na dylatometrze DT 1000. Przedyskutowano przyczyny występowania uszkodzeń matrycy wykonanej z tej stali. W ramach dyskusji przeanalizowano strukturę dyslokacyjną uszkodzonej matrycy, jak i mikrostrukturę warstwy naazotowanej oraz udarność.

1. Introduction

Microstructure of dies has a strong influence on their performance and reliability as far as mechanization and automation of production lines are concerned. Optimal microstructure of dies may be obtained by designing a suitable chemical composition and properly designed heat treatment [1,2]. Usually better properties are obtained in steels with complex chemical composition comparing to steels containing a large amount of one or two elements [3].

Hot working tool steels already at the stage of designing of their chemical composition are anticipated to be in a medium and high tempered state in order to obtain a stable microstructure and thus stabilized properties during work. Nowadays the hot working tool steels have complex chemical composition, contain between 0.25 and 0.6% of C and are characterized by certain kinetics of phase transformations during tempering [4]. Only then, it would be possible to find a suitable heat treatment which results in an optimal combination of mechanical properties and overall performance [5,6].

Main objective of this study was an optimization of dies microstructure made of the 50CrMoV18-30-6 steel and of the microstructure of nitrided layer ensuring a failure-free operation of these tools. The optimization was realized be a deep study of transformations kinetics of super-cooled austenite on the basis of a CCT diagram, dilatometric tests and microstructural investigations (including electron microscopy) of a service-damaged die.

2. Test material

The research was conducted on damaged in-service a die made of 50CrMoV18-30-6 tool steel with a chemical composition presented in table 1 the damaged die is shown in Fig. 1.

^{*} FACULTY OF METALS ENGINEERING AND INDUSTRIAL COMPUTER SCIENCE, AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, 30-059 KRAKÓW, 30 MICKIEWICZA AV., POLAND

TABLE 1 Chemical composition (wt. %) of the investigated steel (die)

С	Mn	Si	Cr	Mo	V
0.5	0.25	0.2	4.50	3.00	0.55



Fig. 1. Damaged die made of 50CrMoVI8-30-6 steel

3. Research results and discussion

Macroscopic observations of the fracture surface show (Fig. 2) that the fracture was of fatigue type, which was radially propagating from the surface of the operating die. It is belivered that the direct cause of the failure was a crack initiated on the surface of the operating die. The surface was nitrided.



Fig. 2. Place of crack initiation

3.1. Nitrided layer

Microscopic observations of subsurface layer revealed morphology and microstructure of nitrided layer (Fig. 3). The thickness of nitrided layer is ca. 200 μ m. It is so thick that the cracks created during operation may

easily obtain a size favouring their further propagation toward the die interior (Fig. 4). Especially in the investigated nitrided layer a bright network of precipitates, most probably of carbonitrides, was observed. The properly nitrated layer should be much thinner than the one found in the damaged die. During operation of the investigated die, its wear results from cracking of nitrated layer rather than due to, e.g. abrasion. However, it is worth to note that the thickness of the nitrided layer was identical in each investigated sample. A presence of white layer on the surface is also unacceptable. A microanalysis performed using a scanning electron microscope (SEM) revealed that the white layer is composed of Fe_4N (ϵ phase). A fact that brittleness of nitrided layer is a main cause of die failure is also confirmed by observations of the fracture using SEM (Fig. 5).



Fig. 3. Nitrided layer. Etched with 2% nital



Fig. 4. Cracks in nitrided layer. Etched with 2% nital



Fig. 5. Cracks in nitrided layer. SEM



Fig. 6. Proper microstructure of steel in as-delivered condition. Etched with 2% nital



3.2. Material in as-delivered condition

It is important to keep in mind that even very faulty microstructure of nitrated layer not necessarily leads to damage of the die if the steel microstructure is optimal with respect to fracture toughness. And vice versa, even very high quality of nitrided layer would not guarantee a long time service of the die when the microstructure of the die core is far from the optimal one. The first condition in order to successfully develop a microstructure of the die by a heat treatment is a suitable microstructure of the steel in as-delivered condition. The steel should be characterized by homogeneously distributed spheroidal carbides in homogeneous ferritic matrix (Fig. 6). However, if steel microstructure before hardening is not homogeneous, then during the heat treatment, phase transformations would proceed with different intensity in different areas and at different temperatures. It may favour generation of stress and make more difficult to choose optimal temperatures of annealing. The 50CrMoV18-30-6 steel requires an special design of soft annealing (spheroidizing). Otherwise the microstructure of the steel delivered to dies manufacturer might be improper (Fig. 7).

Fig. 7. Improper microstructure of steel in as-delivered condition. Etched with 2% nital

3.3. Transformation kinetics of under-cooled austenite

The suitable design of heat treatment should be performed on the basis of the previously prepared CCT diagram. Austenitizing temperature for the preparation of CCT diagram should be selected on the basis of critical points determined from dilatometric tests (Fig. 8) and on the basis of so called hardening series (Fig. 9). This lead to the conclusion that eutectoid transformation of the test steel ends at 895°C. Therefore the temperature of austenitizing should be higher than 895+50°C. What is more, the austenite grain coarsening should be prevented (Fig. 10) by the controlled amount of precipitates of secondary alloy carbides (an electron micrograph of that type of carbides is presented in Figure 11). Hardening from this temperature also ensures relatively high hardness.



Fig. 8. Characteristic points determined on the basis of dilatometric measurements



Fig. 9. Hardening series of test steel



Fig. 10. Microstructure after quenching from: (a) 1030°C, (b) 1050°C, (c) 1070°C, (d) 1100°C



Fig. 11. Secondary carbide in investigated damaged die: (a) bright field image, (b) dark field image, (c) diffraction, (d) diffraction solution. TEM

The analysis of microstructures presented in Figure 10 allows us to state that optimal (safe) austenitizing temperature is 1050°C. For this austenitizing temperature a CCT diagram has been prepared (Fig. 12). The CCT diagram shows that at high cooling rates (5 and 10°C/s) a narrow range of bainitic transformation is present. Most probably it is connected with the beginning of precipitation during cooling, at high temperature, of carbides

on austenite grain boundaries (the range of this precipitation has not been plotted in the CCT diagram due to difficulty of its precise determination). Such precipitates may contribute to lower fracture toughness of the die made of this steel. Therefore the dies made of this steel should be quenched with the highest possible cooling rate.



Fig. 12. CCT diagram of test steel

Correctness of heat treatment proposed by the steel producer has been verified by simulating its run using DT1000 dilatometer. Figure 13 presents dilatograms of individual stages of the heat treatment. A dilatogram presenting a simulation of hardening process is presented in Figure 13a. As one may notice, the martensite start temperature Ms of the test steel is 295° C. A dilatogram of heating to the first tempering temperature, 540° C, is presented in figure 13b. During the heating from as-quenched state three basic transformations may be observed: precipitation of carbides ($80 \div 200^{\circ}$ C), cementite precipitation ($200 \div 370^{\circ}$ C) and partial transformation of retained austenite ($220 \div 280^{\circ}$ C).



Fig. 13. Dilatometric analysis of individual stages of investigated steel heat treatment: a) dilatogram of cooling at the rate of 10° C/s from 1050° C, b) dilatogram of heating at the rate of 10° C/min to 540° C, c) dilatogram of annealing at 540° C, d) dilatogram of heating at the rate of 10° C/min to 560° C, g) dilatogram of annealing at 610° C, f) dilatogram of heating at the rate of 10° C/min to 560° C, g) dilatogram of annealing at 560° C

Figure 13c presents a dilatogram of annealing the steel at 540°C for 3 hours. There is a positive dilatation effect observed during the whole time of the annealing at this temperature. The effect is connected with further transformation of retained austenite. It is partly by a dilatation effect of cementite precipitation. Heating for a second tempering (610°C) produces the dilatogram presented in Figure 13d. A small but well defined effect of ε carbide precipitation from martensite, created as a result of retained austenite transformation, has been recorded. Similarly as during annealing at 610°C, a positive dilatation effect connected with the precipitation of independently nucleating carbides of MC- and M2C-type has been recorded (Fig. 13e). The precipitation of these carbides causes on increase of hardness (secondary hardness).

The progress of heating for the third tempering is presented in Figure 13f. Also in this case, an minute dilatation effect connected with the precipitation of carbides has been recorded. It may be explained by further decomposition of retained austenite during second tempering. During annealing at 560°C a positive dilatation effect connected with further precipitation of independently nucleating carbides or possible transformation of M₂C-type carbides into more stable of M₆C- and M₂₃C₆-type ones has been recorded (Fig. 13g).

3.5. Morphology of carbides as a likely cause of lower fracture toughness

The incorrect morphology of carbide precipitates may be a result of non-optimally performed heat treatment. Precipitates of such carbides are presented in Figure 14. The carbides nucleate on grain boundaries and they grow towards the grain interiors.



Fig. 14. $M_{23}C_6$ carbides on a grain boundary during growing toward the grain interior. TEM

Research performed in this work allowed us to formulate the following conclusions:

- 1. The incorrect morphology of nitrided layer may be one of the main cause of die failure.
- 2. Prior to heat treatment the steel microstructure should be homogeneous (evenly distributed carbides in the ferrite matrix).
- 3. The selected austenitizing temperature should ensure high hardness after quenching together with fine grain microstructure.
- 4. Due to high content of alloy elements forming carbides, especially Mo, hardening of the investigated steel should be performed at the highest possible rate to prevent carbide precipitation on one hand and hardening cracks formation on the other.
- 5. The tempering procedure should minimize the precipitation of carbides on grain boundaries.

Acknowledgements

Project financed by the Ministry of Science and Higher Education, completed under AGH-UST's own research activities no. 10.10.110.855.

The author thanks Prof. J. Pacyna and M.Sc. S. Derlatka for their help in this research.

REFERENCES

- [1] J. R. Davis et al., ASM Handbook 4. Heat Treating, ASM international 1991.
- [2] C. R. Brookes, Principles of the heat treatment of plain carbon and low alloy steels, Materials Park, ASM International, 1999.
- [3] J. Pacyna, A. Jędrzejewska-Strach, M. Strach, The effect of manganese and silicon on the kinetics of phase transformations during tempering - Continuous Heating Transformation (CHT) curves, Journal of Materials Processing Technology 64, 311-318 (1997).
- [4] J. P a c y n a, The effect of retained austenite on the fracture toughness of high speed steels, Steel Research 58, 2, 87-92 (1987).
- [5] P. B a ł a, J. P a c y n a, The kinetics of phase transformations during tempering of the new hot working tool steel designed for a large size forging dies. Steel Research International **79**, 407-413 (2008).
- [6] P. B a ł a, J. P a c y n a, The influence of kinetics phase transformation during tempering on the mechanical properties of HS6-5-2 steel, Journal of Achievements in Materials and Manu-facturing Engineering 28, 123-130 (2008).