Effects of Nb, Ti and V on recrystallization kinetics of austenite in HSLA steels

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The work presents research results of impact of Nb, Ti and V microadditions on recrystallization kinetics and microstructure of newly elaborated steels assigned for production of forged machine parts, using the method of thermomechanical treatment. The study was performed with the use of Gleeble 3800 simulator. In order to determine recrystallization kinetics of plastically deformed austenite, discontinuous compression tests of specimens were done with a given strain at the rate of 10 s$^{-1}$, in a temperature range from 900 to 1100°C, with isothermal holding of samples between successive stages of deformation for 2 to 100 s. Recrystallization kinetics of plastically deformed austenite was described using the Johnson-Mehl-Avrami equation. Performed two-stages compression tests revealed that microadditions introduced into steel considerably influence the kinetics of static recrystallization. Determined time of total recrystallization of austenite, $t_{R}$, in a temperature range from 1100°C to 900°C changes from 100 to 600 s and from 300 to 800 s — for the Ti–V steel and Ti–Nb–V steel, respectively. Executed hot compression tests will contribute to establishing conditions of forging with the method of thermomechanical treatment. 

Key words: HSLA steels, thermomechanical treatment, static recrystallization.

1. INTRODUCTION

HSLA-type (High Strength Low Alloy) microalloyed steels — containing up to 0.3% C and 2% Mn and microadditions with high chemical affinity to N and C, i.e. Nb, Ti and V in the amount of about 0.1%, and sometimes also slightly increased concentration of N and up to 0.005% of B, increasing hardenability — are particularly useful for production of forged parts with fine-grained microstructure using the method of thermomechanical processing. The interaction of microadditions in steel in a solid state depends on their state under conditions of performed plastic working. Microadditions dissolved in a solid solution raise the temperature of recrystallization, with base composition is equal approximately 9 s and increases up to 80 s when adding Mo, Nb and V in a given concentration, and the time of complete recrystallization of austenite increases even more, from about 20 to 2000 s. Broken lines marked in the figure define the probable course of the kinetic curves without the influence of dispersive carbide particles. Inhibiting impact of alloying constituents introduced into steel on the course of recovery and static recrystallization of austenite is particularly effectively noted after decreasing the temperature of plastic deformation and the temperature of isothermal holding to 900°C (Fig. 1b). At such temperature, Mo and a slight portion of introduced V can be found in a solid solution in dissolved state. Nb and the remaining part of V are bounded into NbC and VC or V$_4$C$_3$ carbides, respectively. In this case, the $t_{R,5}$ time necessary to form 50% fraction of austenite recrystallized at such temperature of steel with basic composition is equal approximately 1.5 s and increases up to 80 s when adding Mo, Nb and V in a given concentration, and the time of complete recrystallization of austenite increases even more, from about 20 to 2000 s. Bounded NbC carbides, respectively. In this case, the time of total recrystallization of alloy and unalloyed steels containing microadditions of elements with high chemical affinity to carbon and nitrogen, bounded in dispersive particles of carbides, carbonitrides or nitrides at recrystallization temperature, may be long. For that reason, the $t_{R,5}$ time has a greater significance for technical purpose than $t_{R}$ time [13÷18].

Given data have a considerable significance in designing the technology of hot-working of steels, especially microalloyed steels, with methods of thermomechanical treatment. In addition, performed analysis indicates that apart from the temperature, considerate influence on recovery and static recrystallization kinetics is exerted by chemical and phase composition of steel. The $t_{R}$ time of total recrystallization of alloy and unalloyed steels containing microadditions of elements with high chemical affinity for carbon and nitrogen, bounded in dispersive particles of carbides, carbonitrides or nitrides at recrystallization temperature, may be long. For that reason, the $t_{R,5}$ time has a greater significance for technical purpose than $t_{R}$ time [13÷18].
tests were carried out with the use of Gleeble 3800 device, allowing to compress specimens according to established program. Axisymmetric samples with 10 mm in diameter and 12 mm in length were used for the purpose of research. In order to minimize the effect of friction on the flow curves tantalum foils were used to prevent sticking and graphite foils as a lubricant. Additionally, both surfaces were covered with nickel-based substance. Compression of specimens was realized at a strain rate of 10 s$^{-1}$. To determine kinetics of recrystallization of plasticity deformed austenite, discontinuous compression tests of samples were performed in a temperature range from 900 to 1100°C with isothermal holding of specimens between successive deformations for 2 to 100 s. In order to determine the content of recrystallized phase, the assumption has been made that the increase of the phase volume fraction is connected with changes of yield stress after time counting from the end of previous deformation. The softening kinetics was determined according to the following dependence, the procedure specified in the work [19]:

$$X = (\sigma_1 - \sigma_0)/(\sigma_1 - \sigma_2)$$

where: $\sigma_0$ and $\sigma_1$ – the stress necessary to initiate plastic strain and its value at the moment of its finish in the first stage of deformation, respectively, $\sigma_2$ – the stress necessary to initiate plastic deformation in the second stage of deformation after $\Delta t$ time between those stages. Recrystallization kinetics of plastically deformed austenite was described with the use of the Johnson-Mehl-Avrami equation:

$$y = 1 - \exp(-k\cdot t^n)$$

where: $y$ – fraction of recrystallized austenite after $t$ time, $k$ – constant, $n$ – index exponent.

In order to reveal grains of prior austenite, metallographic observations of samples cooled in water directly after applied strain value and successive etching of metallographic specimens was conducted in OPTON, AXIOVERT 405M light microscope, with magnification ranging from 100 to 800×. Metallographic specimens were prepared according to axis of a sample, in a distance of 1/3 of radius from the centre of sample.

### 3. RESULTS AND DISCUSSION

Conducted research of recrystallization of the A and B steel after two-stage hot compression allowed to determine the influence of testing temperature on the kinetics of thermally activated processes. Discontinuous compression tests of specimens at given strain revealed, according to expectations, that there is a partial and even complete decay of strain hardening between two stages of deformation, depending on the strain temperature and the time of isothermal holding. It’s a consequence of the course of static recovery and static recrystallization.

Typical $\sigma$–$\varepsilon$ curves, registered during compression of samples at a rate of 10 s$^{-1}$, at the temperature of 900°C and 1100°C, in the process of two-stage deformation using isothermal holding for 2, 5, 10, 50 and 100 s, between the first and second deformation of the steel A, are shown in Figure 2. A comparison of $\sigma$–$\varepsilon$ curves in Figure 2a reveals that after the interval of 10 s between the first and second stage of deformation under mentioned conditions, softening of strain hardening is only partial, and after 100 s — almost complete.

The impact of alloying elements, dissolved in a solid solution and the presence of dispersive particles of MX-type interstitial phases on a rate of recovery and mobility of recrystallization front, significantly influences kinetics of static recrystallization of studied steels. This interaction is illustrated as strain hardening curves of phases on a rate of recovery and mobility of recrystallization front, and the presence of dispersive particles of MX-type interstitial compounds.

<table>
<thead>
<tr>
<th>Ti-V (steel A)</th>
<th>Ti-Nb-V (steel B)</th>
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<tbody>
<tr>
<td>C</td>
<td>0.31</td>
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<tr>
<td>Mn</td>
<td>1.45</td>
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<tr>
<td>Si</td>
<td>0.30</td>
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<tr>
<td>P</td>
<td>0.006</td>
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<td>S</td>
<td>0.004</td>
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<td>Cr</td>
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<td>Nb</td>
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<td>Ti</td>
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<tr>
<td>V</td>
<td>0.033</td>
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<tr>
<td>B</td>
<td>0.008</td>
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### 2. EXPERIMENTAL PROCEDURE

The research was performed on newly elaborated microalloyed steels. Chemical composition of steel (Tab. 1) was designed taking into consideration the production of forged machine elements with energy-saving method of thermomechanical processing. Investigated steel melts, weighing 100 kg, were done in VSG-100 type energy-saving method of thermomechanical processing. Investigations of steel melts, weighing 100 kg, were done in VSG-100 type energy-saving method of thermomechanical processing. In order to determine the effect of temperature and time of isothermal holding between two stages of deformation on changes of flow stress, strain hardening and degree of softening, plastometric
It’s a result of decreasing value of self-diffusion coefficient of Fe and coefficients of volume diffusion of alloying constituents along with lowering the temperature as well as influence of their atoms and dispersive particles of interstitial phases of microadditions introduced into steel on the migration rate of recrystallization fronts. In case of the A steel, Mo and V and partially Ti can be found in austenite at the temperature of 1100°C in dissolved state. Interaction of dissolved elements leads to elongation of recovery time and decrease of recrystallization rate due to segregation of those atoms in the deformation field of dislocations and on recrystallization fronts, causing decrease of their mobility.

The greatest influence on elongation of times of recovery and recrystallization is exerted by contribution of segregation of dissolved atoms and dispersive particles of interstitial phases. As shown in Figure 3, the time \( t_{0.5} \) necessary to form 50% fraction of recrystallized austenite for the A steel at the temperature of 1100°C is equal 6 s and increases to about 30 s together with decrease of the compression temperature to 900°C. Time of total recrystallization of austenite, \( t_{R} \), which varies from 100 to about 600 s in investigated temperature range, drags even more.

Strain hardening curves for the B steel containing 0.28% C, 1.41% Mn, 0.22% Mo and microadditions of Nb, Ti and V in the amount of 0.027% and 0.028% and 0.019%, respectively, are presented in Figure 3 (dotted line). Data shown in the figure indicates that inhibiting interaction of alloying elements introduced into steel on the course of recovery and static recrystallization of austenite is particularly effectively noted after decreasing the temperature of plastic deformation and the temperature of isothermal holding to 900°C. At this temperature, there is Mo and microaddition of vanadium present in a solid solution in dissolved state. In turn, microadditions of Nb and Ti are completely bounded into NbC, TiC, TiN and (Ti, Nb)(C, N) at this temperature.

The shape of analysed kinetic curves indicates the possibility of describing them in accordance with the model of recrystallization after hot deformation, taking into consideration the process of static recovery, metadynamic recrystallization and static recrystallization. Fraction of metadynamic recrystallization to the decrease of strain hardening after deformation of the A steel, realized at the temperature of 1100°C is possible because of applied value of strain equal \( \varepsilon = 0.2 \), close to the \( \varepsilon_{cd} \) value, required for initiation of dynamic recrystallization. Applied degree of deformation, at lower strain temperature, was lower than required for initiation of the process of dynamic recrystallization. Nevertheless, after deformation of the A steel at the temperature of 900°C, at the rate of 10 s\(^{-1}\), decrease of strain hardening is caused by the process of static recovery and static recrystallization, and at the temperature of 1000 and 1100°C — by the process of static recovery, metadynamic recrystallization and static recrystallization. Whereas, in the B steel, deformed at the temperature of 900 and 1000°C, the decrease of strain hardening is a result of the course of static recovery and static recrystallization, and deformed at 1100°C, it’s controlled by the course of metadynamic and static recrystallization.

Furthermore, conducted discontinuous compression tests of investigated steels in the two-stage deformation applying isothermal holding for the time ranging from 2 to 100 s revealed that kinetics curves of recrystallization of plastically deformed austenite of the B steel are clearly displaced towards right in relation to kinetics curves of plastically deformed austenite of the A steel. It should be explained with interaction of Nb microaddition, which presence in the B steel has a great influence on the decrease of rate of thermally activated processes. The beginning of precipitation of NbC carbides for equilibrium conditions occurs at the temperature of about 1135°C [21]. It means, that certain portion of niobium is bounded into dispersive particles of NbC carbides, formed on dislocations in plastically deformed austenite, already at the temperature of

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**Fig. 2.** The \( \sigma-\varepsilon \) curves registered during double-hit compression test of A steel samples at the temperature of 900°C (a) and 1100°C (b) at a rate of 10 s\(^{-1}\) with the use of intervals between successive stages of deformation equal 2, 5, 10, 50 and 100 s

**Rys. 2.** Krzywe \( \sigma-\varepsilon \) zarejestrowane podczas dwuetapowego ściskania na gorąco próbek ze stali A w temperaturze 900°C (a) i 1100°C (b) z prędkością 10 s\(^{-1}\) z zastosowaniem przerw między kolejnymi etapami odkształce- nia wynoszących 2, 5, 10, 50 i 100 s

**Fig. 3.** Effect of the test temperature on the static recrystallization kinetics of plastically deformed of A and B steel in hot compression test

**Rys. 3.** Wpływ temperatury badania na kinetykę rekrywalizacji statycznej stali A i B odkształconych plastycznie w próbie ściskania na gorąco
1100°C. The fraction of NbC carbides in a solid solution increases along with temperature decrease, and niobium is bounded into NbC at the temperature of 900°C. The detailed informations concern the process of dispersion particles precipitation of the MX-type interstitial phase and their influence on austenite microstructure of steels studied has been shown in works [20, 21].

Experimentally determined values of the \( t_{0.5} \) time for the A and B steel reveal good correspondence with the model equations, written as [22, 23]:
- for A steel: \( t_{0.5} = 6.767 \times 10^{-10} \cdot \varepsilon^4 \cdot \varepsilon^4 \cdot \delta \cdot \exp(166000/(RT)) \) (3)
- for B steel: \( t_{0.5} = 2.52 \times 10^{-19} \cdot \varepsilon^4 \cdot \delta^2 \cdot \exp(325000/(RT)) \) (4)

Comparison of model and experimental values of the \( t_{0.5} \) time for the A and B steel is presented in Figure 4.

Large austenite grains with average diameter of 64 μm, after static recovery occurring in the time equal 2 s, were revealed in the microstructure of the A steel, deformed in two stages at the temperature of 900°C. After isothermal holding at the temperature of 900°C for 5 s there is a decrease of the rate of the course of thermally activated processes, connected mostly likely to formation of nuclei of static recrystallization.

Isothermal holding of the A steel specimens at such temperature for 10 s leads to the initial stage of static recrystallization, wherein in these conditions the degree of softening (decay of strain hardening) is equal \( X = 0.32 \). Increase of isothermal holding time up to 100 s (\( X = 0.83 \)) results in grain growth of austenite. Distinct refinement of primary austenite grain in comparison to the initial state, caused probably by partial course of dynamic recrystallization during deformation and the course of metadynamic and static recrystallization after deformation was observed after two-stage compression of the A steel at the rate of 10 s\(^{-1}\), at the temperature of 1000 and 1100°C. Isothermal holding after deformation of the A steel specimens at the temperature of 1000°C for 2 and 5 s leads to obtaining 23% and 32% fraction of recrystallized austenite, respectively and the size of the phase of 32 μm and 22 μm, respectively. Slowing down of the process of decay of hardening is observed after isothermal holding at the temperature of 1000°C for 10 s, what is connected with formation of nuclei of static recrystallization. Austenite grain size after isothermal holding of specimens for 10 s is equal approximately 16 μm, and the recrystallized fraction is equal to 50%. The progress of recrystallization process and growth of γ phase grains is observed for the time of isothermal holding equal 50 and 100 s. Isothermal holding of samples for 50 s leads to obtaining 84% fraction of recrystallization and equivalent diameter of austenite grains of approx. 35 μm. Increase of time up to 100 s results in obtaining microstructure characterized with 95% fraction of recrystallization and average diameter of γ phase grain size equal 78 μm. Decrease of hardening of the A steel after double-hit compression at the temperature of 1100°C is determined by the course of dynamic recrystallization after isothermal holding of samples at this temperature for 2 and 5 s, and successively, by the course of static recrystallization for the time of holding equal to 10, 50 and 100 s.

The size of primary grains of austenite varies from about 27 μm for holding time of 2 s, to about 63 μm for holding time equal 100 s. In the result of two-stage compression of the B steel at the temperature of 900°C, at the rate of 10 s\(^{-1}\), diversified grain size of primary austenite, in a range from 25 μm to 70 μm, was obtained in a consequence of static recovery and static recrystallization. Two-stage deformation of the B steel at the temperature of 1000°C with the use of isothermal holding for the time equal from 2 s to 100 s caused a decrease of strain hardening as a result of the course of static recovery and static recrystallization. Two-stage deformation of the B steel at the temperature of 1000°C with the use of isothermal holding for the time equal from 2 s to 100 s caused a decrease of strain hardening as a result of the course of static recovery and static recrystallization. Two-stage deformation of the B steel at the temperature of 1000°C with the use of isothermal holding for the time equal from 2 s to 100 s caused a decrease of strain hardening as a result of the course of static recovery and static recrystallization. Two-stage deformation of the B steel at the temperature of 1000°C with the use of isothermal holding for the time equal from 2 s to 100 s caused a decrease of strain hardening as a result of the course of static recovery and static recrystallization. Two-stage deformation of the B steel at the temperature of 1000°C with the use of isothermal holding for the time equal from 2 s to 100 s caused a decrease of strain hardening as a result of the course of static recovery and static recrystallization.

4. CONCLUSIONS

Performed two-stage compression tests revealed, that apart from the temperature, considerable influence on kinetics of recovery and static recrystallization is exerted by chemical and phase composition of steel after hot-working is concluded and that the \( t_{0.5} \) time of total recrystallization of steel containing microadditions of elements with high chemical affinity for carbon and nitrogen, bounded in dispersive particles of carbides, carbonitrides or nitrides at recrystallization temperature, is long. Determined time of total recrystallization of austenite, \( t_{0.5} \), in a temperature range from 1100°C to 900°C changes from 100 to 600 s and from 300 to 800 s — for the Ti-V steel and Ti-Nb–V steel, respectively. It means that the complete recrystallization of austenite requires long times, unacceptable in a production process of forgings. Therefore, the \( t_{0.5} \) time has a greater meaning than \( t_{0.5} \) for technological purposes. Moreover, considerable deformation at high rate and short intervals for moving produced parts from one die to another do not create convenient conditions for the course of static recrystallization, allowing grain refinement of austenite. Hence, forgings should be isothermally held at a temperature of forging finish prior to hardening for the time necessary to form about 50% fraction of recrystallized austenite, which for the
A steel is equal 30 s, and 60 s for the Nb-containing steel. Lower rate of static thermally activated processes in the B steel in relation to the A steel is a result of the presence of Nb microaddition, which being formed on dislocations during plastic deformation in a form of dispersive NbC and (Ti, Nb)(C, N) particles, slows down the course of recovery and dynamic recrystallization, and after hot-working is done, decreases the rate of recovery and static recrystallization and limits grain growth of recrystallized austenite.

REFERENCES


Oddziaływanie Nb, Ti i V na kinetykę rekrystalizacji austenitu w stalach typu HSLA
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1. CEL PRACY
W pracy przedstawiono wyniki badań oddziaływania mikrododatków Nb, Ti i V na kinetykę rekrystalizacji i strukturę nowo opracowanych stali typu HSLA przeznaczonych do wytwarzania kutych elementów maszyn metodą obróbki cieplno-plastycznej.

2. MATERIAŁ I METODYKA BADAN
Badania przeprowadzono na dwóch stalach mikrostopowych typu HSLA o składzie chemicznym zestawionym w tabeli 1. Wytopy stali o masie 100 kg wykonano w laboratoryjnym, próżniowym piecu indukcyjnym typu VSG-100S firmy PVA TePla AG. W celu określania temperatury odkładania oraz czasu wytrymania izotermicznego między dwoma etapami odkładania na zmiany naprężenia uplastyczniającego, stopień mięknięcia i strukturę stali wykonano badania plastometryczne za pomocą urządzenia Gleeble 3800, będącego na wyposażeniu Instytutu Metalurgii Żelaza. Do badań użyto próbek osiowo-symetrycznych o średnicy 10 mm i długości 12 mm. W celu wyznaczenia kinetyki rekrystalizacji austenitu odkształconego plastycznie wykonano przerywane próby ścianania próbek na gorąco na pozycji 45° na mikroskopie świetlnym AXIOVERT 405M firmy OPTON w zakresie powiększenia od 100 do 800×.

3. WYNIKI I ICH DISKUSJA
Przeprowadzone badania rekrystalizacji obydwóch stali po dwuetapowym ściananiu na gorąco pozwoliły na określenie temperatury odkładania na kinetykę procesów aktywowanych cieplnie. Przerywane próby ścianania próbek wykazały zgodnie z oczekiwaniami, że podczas wytrymania izotermicznego między dwoma etapami odkładania następuje częściej niż w przypadku stabilizacji, a nawet całkowity zanik umocnienia odkładanowego, zależnie od temperatury odkładania oraz czasu wytrymania izotermicznego. Wyniki badań kinetyki zaniku umocnienia odkładanowego badanych stali zestawiono na rysunku 3. Przebieg analizowanych krzywych kinetyki wskazuje na możliwość opisania ich zgodnie z modelem rekrystalizacji po odkładaniu na gorąco, uwzględniającym proces zdrowienia statycznego, rekrystalizacji metadynamicznej i rekrystalizacji statycznej. Po odkładaniu plastycznym stali A w temperaturze 900°C z prędkością 10 s⁻¹, za zmniejszenie umocnienia odkładanowego jest odpowiedzialny proces zdrowienia statycznego i rekrystalizacji statycznej, a w temperaturze 1000 i 1100°C proces zdrowienia statycznego, rekrystalizacji metadynamicznej i rekrystalizacji statycznej. Natomiast w stali B odkładanej w temperaturze 900 i 1000°C zmniejszenie umocnienia odkładanowego jest wynikiem przebiegu zdrowienia statycznego i rekrystalizacji statycznej, a odkładanej plastycznie w temperaturze 1100°C jest kontrolowany przebiegem rekrystalizacji metadynamicznej i statycznej. Przeprowadzone próby ścianania próbek badanych stali wykazały, że krzywe kinetyki rekrystalizacji austenitu odkładanowego plastycznego stali B są wyraźnie przesunięte w prawo w porównaniu z krzywymi kinetyki rekrystalizacji austenitu odkładanowego plastycznego stali A. Należy do tymATCHOWANIE MIODEY ODSYLVANCE, PRAWO W POROWNaniu, Z KREDYWNYMI KINETYKỳ, REKRZYSTALIZACJÌ, UDZELOKCU_mAŃSTCY, NASTOMIÀT W STALÌ B OA KOSIDEŁU NA GORÀÇO, POSŁUZÀÀ DO USTÄLENIA WARUNKÓW IAKLAUACJÓN, A W TEMPERATURZE 900°C CAŁY NIÓBZ JEST ZWINIANY W WĘGŁIKI I WĘGŁIKOAZOTKI.

4. PODSUMOWANIE
Przeprowadzone dwuetapowe próby ścianania wykazały, że oprócz temperatury wpływ na kinetykę zdrowienia i rekrystalizacji statycznej wywiera skład chemiczny i fazowy stali po zakończeniu odkładania plastycznego na gorąco. Stwierdzono, że czas całkowitej rekrystalizacji \( t_{50} \) stali zawierających mikrododatki pierwiastków o dużym chemicznym powinowactwie do węgla i azotu, związanych w temperaturze rekrystalizacji w dyspersyjne cząstki węglików NbC, tworzących się na dyslokacjach w austenicie odkształconym plastycznie. Wraz z obniżeniem temperatury udział w roztworze stałym faz międzywęzkowych węglików, węglikoazotków lub azotków jest długiego. Wyznaczony czas całkowitej rekrystalizacji austenitu \( t_{50} \) w zakresie temperatury od 1100°C do 900°C zmienia się od 100 do 600 s i od 300 do 800 s, odpowiednio dla stali A i B. Oznacza to, że całkowity przebieg rekrystalizacji austenitu badanych stali wymaga długiego czasu, nie do zaakceptowania w procesie produkcyjnym odkuwek. Dlatego dla celów technicznych większe znaczenie niż \( t_{50} \) ma czas \( t_{5} \). Ponadto znaczące odkształcenia z dużą szybkością oraz krótkotrwałe przerwy na przeniesienie wytwarzanego elementu z jednego do drugiego wykroju matrycy nie stwarzają dogodnych warunków do przebiegu rekrystalizacji statycznej, umożliwiającej rozdrobnienie ziaren austenitu. Dlatego odkuwki przed hartowaniem należy wytrzymać izotermicznie w temperaturze końca kucia przez czas potrzebny do utworzenia 50% frakcji austenitu zrekrytalizowanego, który dla stali A wynosi 30 s, a dla stali B wynosi 60 s. Przeprowadzone próby ścianania na gorąco pozwoliły do ustalenia warunków kucia stali mikrostopowych metodą obróbki cieplno-plastycznej.