Petr Kratochvíl; Ladislav Havela; A. Svobodová; Josef Pacák; J. Tomeš Static recrystallization processes in high alloyed seamless tubes of AISI 321 Steel after the hot formation

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Static Recrystallization Processes in High Alloyed of AISI 321 Steel after the Hot Formation	yed Seamless Tubes	
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Inohomogeneous deformation takes place during hot working of seamless tubes of AISI 321 steel by pilgrim milling. The restystallization process is therefore not completed in the whole cross section of the tube. It is shown how to reach the total recrystallization by improving both the composition of the steel and the whole thermal processing.

Při poutnickém válcování silnostěnných trubek za horka dochází k nehomogenní deformaci ve stěně. Následné rekrystalizační žíhání je neúčinné. Je ukázáno jak dosáhnout úplné rekrystalizace na základě úpravy složení oceli a postupu tepelného zpracování.

При прокате тольстостенных трубок при повышенной температуре происходит негомогенная деформация в стенках. Последующий рекриссталлизационный отжиг не дает эффекта. Показано, как достичь полной рекристаллизации на основе изменения состава стали и процесса тепловой обработки.

1. Introduction

Very detailed study of the mechanical properties of AISI 321 steel used in nuclear power stations was completed recently [1, 2]. It is a purpose of the present paper to use the knowledge on the physics of high temperature deformation to improve the structure of seamless tubes with great cross sections by the change in thermomechanical treatment. Large regions detected by the ultrasonic methods appear very often after the hot forming of great pieces. The substructure typical for the hot deformation process was observed in these regions. It is supposed that in these regions under given conditions not enough stored energy accumulates to initiate the static recrystallization during subsequent final annealing.

The proportion of defect regions should be suppressed to minimum or even to zero. The main features of our experiment are:

(i) the study of the effect of the content of precipitate forming solutes (Ti, C);

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- (ii) the study of the effect of the change of static annealing conditions, i.e. of the annealing temperature T_a , the annealing time t_a ;
- (iii) the study of the effect of the forming conditions, namely of the wroking temperature T_w .

The experimental conditions were chosen so as to approximate the technology of great diameter tubes as closely as possible.

2. The technology used for the production of great diameter seamless tubes

The tubes with a diameter d > 200 mm were produced by hot pilgrim rolling process. The typical composition of austenitic stainless steel AISI 321 is as follows (wt.%):

C	0.06
Mn	1.50
Si	0.80
Cr	18.00
Ni	10.50
P	0.03
S	0.02
Ti min. 5 × wt.% C	0.60
N	0.05
Co	0.02
Cu	0.30

The typical thermomechanical processing can be summarized as follows, see also the scheme in Fig. 1:

- (i) The prisms (285 mm) are heated in a rotary furnace to 1280 °C and pierced in the hydraulic press to obtain "a tube" (Ø 340/180). The average deformation during this process is 12%.
- (ii) The cylinders are heated again to 1230 °C and elongated in a double rollpiercing mill to Ø 330/195. Here the deformation is ~11%.
- (iii) The later process is immediately followed by pilger milling to $\emptyset 223/32$ during which the average deformation is ~82%.

It was shown that the last stage is the determining one for the substructure. That is followed by the solution treatment. The temperature during the pilgrim milling was estimated to be between $1050 \,^{\circ}$ C and $1020 \,^{\circ}$ C on the outer surface of the tube The inner surface of the tube is substantially cooler due to the contact with the cold mandrel rod.

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1. The scheme of the thermomechanical processing of seamless tubes.

An area ranging to 5-10 mm from the inner surface contains large not recrystallized grains after the solution annealing during which the static recrystallization should take place. The typical substructure is shown in Fig. 2. The substructure with elongated not recrystallized grains is typical for the hot formation process i.e. for dynamic recovery (DRV) or dynamic recrystallization (DRX).

3. Experimental and discussion

3.1. Character of the deformation process in conditions comparable to pilgrim rolling

The samples used for the deformation in tension were taken from the tube before pilgrim rolling, i.e. after the elongation process, as shown in Fig. 3. The gauge length of the sample was 55 mm and their diameter 6 mm. They were deformed with a strain rate comparable to the pilgrim rolling process $\dot{\varepsilon} = 1.3 \times 10^{-1} \text{ s}^{-1}$ at several $T_{\rm w}$. The testing machine INSTRON TT 1185 was used. The true stress – true strain curves are given in Fig. 4. All the curves are typical for DRV. For the deformation at 850 °C the cell structure was typical (Fig. 5). The subgrains start to form and do persist through the deformation at 900 °C as a characteristic feature (Fig. 6). During the deformation at 950 and 1000 °C the recrystallized grains are also observed



2. The typical substructure with faulted zones at the inner surface of the tube. The view the $100 \times$ magnification showing the not recrystallized grains elongated in the pilgrim milling direction.

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(Fig. 7). Their origin is to be found in the metadynamic processes. The only conclusion is, that the DRX takes place. Such processes are a major phenomenon during the deformation at 1050 $^{\circ}$ C (Fig. 8).



3. The position of the samples used for the tension deformation, the scheme in the half-finished product (i.e. before elongation and pilgrim milling).



4. The stress-strain curves for the samples in Fig. 3 deformed at different temperatures.









8. Substructure of the material after the deformation at $1100 \,^{\circ}$ C.

3.2. The effect of static annealing conditions

3.2.1. Model experiment

The pilgrim rolling was simulated by hot compression of small cylinders ($\emptyset = 8 \text{ mm}$, length l = 16 mm). Two subsequent deformations:

- 1. 30% at 1050 °C and
- 2. 20% at 950 °C

are near to the conditions during elongation and pilgrim processes resp. in the neighbourhood of the inner surface of the tube, where the deformation may be also less than this value. The strain rate used during this experiment was also $\dot{\varepsilon} = 1.3 \times 10^{-1} \, \text{s}^{-1}$. This deformation process was followed by annealing at 1050 °C for 30 minutes, which is typical for the situation in tube production.



TIME-STRAIN

9. The scheme of the thermomechanical processing: After two hot deformations at different temperatures the annealing temperature is reached either directly — mode 3 — or after cooling the sample to the room temperature — mode 2 —. Mode 1 — is a sample which was not annealed after cooling to the room temperature.

The value of the ratio of the recrystallized volume X_R was measured for different thermal treatments (see Fig. 9). The annealing temperature T_a is reached:

(i) immediately after completing the hot formation; mode 3 in Fig. 10;

(ii) after cooling the sample to the room temperature; mode 2 in Fig. 10.

The value of $X_{\rm R}$ for the sample, which was not annealed is also given in Fig. 10 (mode 1).

It is obvious from Fig. 10, that the value of X_{R} depends very pronouncely on the



The recrystallized ratio X_R for different modes, 1, 2 and 3. Points (●) are for the steel with higher Ti and C contents and points (○) for that with lower content of Ti and C.

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11. The time development of X_R as obtained by fitting of the experimental data to the eq. (1). The experimental data are included. The plots are valid only for the steel with higher concentration of Ti and C. For the steel with other Ti and C contents new plots are to be constructed for the use in the tube production.

way, how the annealing temperature T_a is reached. Leaving out the cooling to the room temperature before annealing we are able to suppress the formation of titanium carbonitride on cooling. The efficiency of such a change in thermal processing was verified during the free forging of great pieces.

This was also proved by repeating the same experiments in the steel with lower contents of Ti and C; namely $c_{\text{Ti}} = 0.30$; $c_{\text{C}} = 0.03$. The result is seen in Fig. 10: The fully recrystallized state is reached with such a material independently on the way how T_{a} is reached. There might be a disadvantage in decreasing the value of the yield stress in such a modified material.

3.2.2. Recrystallization process

A detailed investigation of the recrystallization process was undertaken to optimize the annealing conditions (T_a, t_a) during production of seamless tubes. This included measurement of X_R as a function of T_a and t_a . The values in Table I and in Fig. 11 are related to the inner surface of the tube which is the "dangerous" region. The structure was observed both in the longitudinal and transversal cross sections. The samples were cut from the tube cooled to room temperature after pilgrim rolling, i.e. before the recrystallization annealing.

$t_{a}(\min)$	$T_{a}(^{\circ}C)$	1060	1080	1100	1140
0		19			
30		33	42	70	93
60			60	90	
70		48			
120		71	83		
180		70			
220				93	
250		91			
400		95			
480		. 96			

Table I

The values from Table I are given in Fig. 11 and fitted by the "recrystallization curve" [3]:

(1)
$$X_{\mathbf{R}} = 1 - \exp\left[-\ln 2\left(t/t_{0.5}\right)\right],$$

where $t_{0.5}$ is the time t_a , for which $X_R = 0.5$. Thus it is possible to predict the effect

of both T_a and t_a and to choose the proper annealing conditions for the technology. It is, nevertheless, necessary to repeat the described procedure if the composition of the stainless steel is changed e.g. for material containing less Ti and C.

The value of d_{G} was measured so as to prevent the unwanted growth of the grain size (mostly during eventual secondary recrystallization). The data are in Table II.

The grain size $d_G(\mu m)$ for some values of T_a and t_a					
$t_{a}(\min)$	$T_{a}(^{\circ}C)$	1060	1080	1100	1140
30			19	16	75
60		18	18	18	
120		15	22		
480		19	26		

Table II The grain size $d_{\rm G}$ (µm) for some values of $T_{\rm a}$ and $t_{\rm a}$

The tables I and II enable to choose such a thermomechanical processing so as to obtain the recrystallized grains in the whole volume of the product. With respect to the above described effect of the reduction of Ti and C contents on the value of $X_{\rm R}$, the same experiment as described by Table I and II was performed with reduced Ti and C contents, i.e. $c_{\rm Ti} = 0.3$ and $c_{\rm C} = 0.03$. The result of this experiment is very promissing for the production: After 30 min. annealing even at 1040 and 1100 °C the recrystallized volumes (i.e. at the inner surface of the tube) were 95 and 100% respectively. The grain size after this treatment was also acceptable: 13 and 28 μ m resp.

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