KINETICS OF GRAIN GROWTH AND RECRYSTALLIZATION DURING FORMING MODES FOR PROCESSING OF STEEL SA 508

Petr ZUNA\(^1\), Jakub HORNÍK\(^1\), Jaroslav MÁLEK\(^1\), František JANDOŠ\(^2\)

\(^1\)CTU in Prague, Faculty of Mechanical Engineering, Technická 4, 166 07 Praha 6, petr.zuna@fs.cvut.cz
\(^2\)PILSEN STEEL s.r.o., Tylova 2090/1, 301 00 Plzeň-Jižní Předměstí

Abstract
Within the optimization of material processing technology for heavy forgings made from the steel SA 508 for power industry (especially shafts), kinetics of grain growth, recrystallization and precipitation processes in order to prevent the occurrence of coarse-grained structures were evaluated. In the typical range of forging temperatures of 850 °C - 1250 °C the austenite grain coarsens significantly above a temperature of 1100 °C. Evaluation of recrystallization processes shows significant inhibition of dynamic and consequently postdynamic recrystallization after deformation of 60% at temperature 850 °C. With increasing deformation temperature the contribution of dynamic and postdynamic recrystallization grows. Particles present in the microstructure evaluated by TEM are mostly M\(_3\)C type. The optimization of material processing technology forged steel SA 508 for power supply applications has been evaluated and grain growth kinetics of precipitation processes in order to prevent undesirable coarse-grained structures.

Keywords:
microstructure, grainsize, recrystallization, precipitation

1. INTRODUCTION
The work deals with the influence of heating conditions and deformation on the kinetics of austenite grain growth of steel SA-508. This work is a part of the research motivated by efforts to optimize the processing conditions to obtain fine-grained structure during the manufacture of wind turbine rotors by free forgings. Production of heavy forgings is specific and very expensive. Using the wrong technological processes, as in the production of ingot and during his subsequent treatment by forming, together with other factors could lead to production of non-conforming product, which entails the necessity of repeated production and therefore considerable economic losses. The common defects occurring during forming of rotors include unwanted local grain coarsening, which can occur as the presence of casting defects in the ingot, the temperature distribution and the inhomogeneity of deformation. The paper dealt with the issue of the deformed state of the material, recrystallization processes and the factors influencing them and possibly occurring during hot forming [1-4]. Objective of the experiment focuses on the laboratory evaluation of kinetics of austenite grain growth depending on the temperature of forming and recrystallization behavior of steels in the range of typical forging temperatures.

2. EXPERIMENTAL
Experimental material was supplied by PILSEN STEEL s.r.o. in the form of machined forging made from steel SA-508 Grade C1.1 (20MnMoNi5-5).

Results of the analysis of chemical composition of evaluated steel are shown in Tab. 1.

Microstructure of the steel was evaluated in the as-delivered state. To evaluate the kinetics of austenite grain growth, the temperature range corresponding to the typical range of temperatures including final forging steps (850, 900, 1000, 1100, 1200, 1250 °C) was chosen. Samples were heat treated in the filling of corundum and crushed coke, restricting decarburization. Heating-up and cooling curves of backfill, furnace and samples were collected for to ensure repeatability of experiments. Holding times at the temperature
were 15 and 60 minutes. Dwell time of 15 min was selected for laboratory processing with minimal decarburization, whereas 60 minutes dwell time better simulates real forging conditions. Samples of dimensions of 15 x 15 x 15 mm were cooled in water after the treatment allowing austenitic grain evaluation.

**Table 1** Chemical composition of evaluated steel SA 508 [wt. %]

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>Co</th>
<th>Ti</th>
<th>V</th>
<th>Al</th>
<th>Nb</th>
<th>B</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.18</td>
<td>1.27</td>
<td>0.27</td>
<td>0.005</td>
<td>0.001</td>
<td>0.07</td>
<td>0.64</td>
<td>0.48</td>
<td>0.03</td>
<td>0.005</td>
<td>0.002</td>
<td>0.010</td>
<td>0.029</td>
<td>0</td>
<td>0.0003</td>
<td>0.004</td>
</tr>
</tbody>
</table>

Cylinders with a diameter of 8 mm and a height of 15 mm were used for the purpose of recrystallization behavior evaluation. The laboratory upsetting was applied using gravity falling hammer weighing 11.6 kg and with a lines height of 3500 mm. Ram impact velocity is 8.3 m.s\(^{-1}\). In case of a sample with a height of 15 mm the equivalent strain rate is approximately 5.5 \(10^2\) s\(^{-1}\). The required deformation of the samples was achieved by inserting a stopper. All the cylinders were deformed to 60 % of their original height. Handling time of the ample from the final deformation to cooling in water was 2 seconds.

Samples were processed using the same austenitizing temperature of 1200 °C for 15 min and air cooling at deformation temperature afterwards. Subsequently, 15 minutes equalizing dwell was performed at the deformation temperature. The samples were deformed by upsetting. Part of them was immediately cooled down in water for the purpose of the dynamic, respectively metadynamic recrystallization evaluation. The other samples were put back into furnace for 60 s, 180 s and 300 s and subsequently cooled down in water. These were used for the purpose of static recrystallization evaluation. The deformation temperature was 850 °C or 1100 °C.

The microstructure was evaluated by light microscopy and scanning electron microscopy combined with EDS microanalysis. A more detailed analysis of the particles was performed using TEM with EDS microanalysis at collodion and carbon extraction replicas. Samples were etched in NITAL and Villela-Bain agents or in hot picric acid reagent in solution with surfactant.

### 3. RESULTS AND DISCUSSION

#### 3.1 As delivered state

The microstructure of forgings in as-delivered state is homogeneous, predominantly bainitic (Fig. 1). The size of the original equiaxed austenite grain is from 50 to 70 \(\mu\)m. Distribution and shape of carbides was evaluated using SEM and subsequently TEM on carbon and collodion replicas (Fig. 2).

![Fig. 1 Microstructure- as delivered state, LM](image1.png)

![Fig. 2 As delivered state, TEM, collodion replica](image2.png)
Carbides are distributed mainly at the boundaries of ferritic needles (Fig. 2). Diffraction images examination complemented by EDS microanalysis of particles shows that the majority of present particles is of the M₂C or M₃C type. Iron makes a primary contribution, followed by manganese and molybdenum. Typical microalloying elements like Ti and V, together with Al were detected in small quantities in M₃C particles and sporadically in the form of separate carbides, nitrides or carbonitrides.

3.2 Kinetics of austenite grain growth

The results of measurements of grain size after annealing at the defined temperatures with the dwell time at the temperature of 15 or 60 minutes are graphically presented in Fig. 3. From the results it is clear that the grain size does not change significantly up to temperatures of 1000 °C. In case of dwell time 15 minutes, grain growth does not take place even at a temperature of 1100 °C. Significant grain coarsening was observed between the temperatures of 1100 °C and 1250 °C. At a temperature of 1250 °C the grain sizes in case of both holding times are similar.

Microstructure of samples austenitized at 900 °C and cooled in water contains a considerable amount of carbide particles at grain boundaries and between the martensite needles (Fig. 4). The grain does not coarsen at this temperature; the particles may effectively inhibit its growth. After austenitizing at 1250 °C, the quantity and particle size are reduced considerably (Fig. 5). Their inhibiting effect decreases, the boundaries can migrate more easily and austenitic grain becomes coarser. A more detailed analysis and particle identification was performed by transmission electron microscopy on extraction replicas (Fig. 6). Using the electron diffraction patterns the particles of M₂C and M₃C (Fe, Mn, Mo), sporadically TiC and AlN type were identified.
3.3 Recrystallization behavior

The fractions of recrystallized volume of samples after deformation at 850 °C and 1100 °C are shown in Table 2. Kinetics of recrystallization plotted in Avrami’s coordinates (Fig. 7) shows a different behavior in case of both temperatures. At a deformation temperature of 1100 °C, dynamic or postdynamic recrystallization of 70% of the volume takes place. After the dwelltime of 180 s at deformation temperature 1100 C the process of recrystallization is completed. Deformation temperature of 850 °C leads to a dynamic (postdynamic) recrystallization only in 4% of volume. The rate of recrystallization increases after 60 s hold with a typical value of kinetic exponent for static recrystallization $n \approx 2$. After the dwell of 180 s at the deformation temperature 850 °C the recrystallized volume fraction reaches 60%. Further dwell of 300 s at the deformation temperature leads to recrystallization of 80% volume. Furthermore, grain size was evaluated in samples where at least 50% of volume was recrystallized. Evaluation was performed in the central region where homogeneous deformation takes effect and in areas of possible critical deformation near the cylinders ends (Table 2). The real deformation (60 - 65)% was measured in the homogeneous region. Deformation of (7 – 10)% was measured in area of the hindered deformation (dead-metal zone) [5]. This value is close to the point where critical deformation occurs and may lead to grain coarsening. In case of used austenitization temperature of 1200 °C the estimated grain size is 380 μm. Recrystallized grain size in the area of homogeneous deformation after upsetting at the temperature of 1200 °C is 49 μm. In the area
of dead zone with deformation (7 - 10) % the recrystallized grainsize reaches 286 μm. Values of grain size corresponding to deformation temperatures 1100 °C and 850 °C are shown in Tab. 2.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1200</td>
<td>850</td>
<td>2</td>
<td>4</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>1200</td>
<td>850</td>
<td>60</td>
<td>7</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>1200</td>
<td>850</td>
<td>180</td>
<td>60</td>
<td>64</td>
<td>204</td>
<td>-</td>
</tr>
<tr>
<td>1200</td>
<td>850</td>
<td>300</td>
<td>80</td>
<td>72</td>
<td>248</td>
<td>-</td>
</tr>
<tr>
<td>1200</td>
<td>1100</td>
<td>2</td>
<td>70</td>
<td>55</td>
<td>212</td>
<td>-</td>
</tr>
<tr>
<td>1200</td>
<td>1100</td>
<td>60</td>
<td>90</td>
<td>80</td>
<td>188</td>
<td>-</td>
</tr>
<tr>
<td>1200</td>
<td>1100</td>
<td>180</td>
<td>99</td>
<td>96</td>
<td>259</td>
<td>-</td>
</tr>
<tr>
<td>1200</td>
<td>1100</td>
<td>300</td>
<td>99</td>
<td>140</td>
<td>313</td>
<td>-</td>
</tr>
</tbody>
</table>

Transmission electron microscopy was used on selected samples. Fig. 8 documents the microstructure and distribution of particles in samples austenitized at 1200 °C and deformed at the temperature of 850 °C and 1000 °C.

Particles existing in the microstructure have predominantly spherical or cubic shape. The particle size does not exceed 0.5 μm. They are located at the austenitic grain boundaries or between martensitic needles in lower quantity. With an increased deformation temperature, the quantity of fine particles present on the grain boundaries decreases (Fig 8).
EDS analysis of chemical composition shows that most of the observed particles are of $M_2C$ or $M_3C$ type with predomination of Fe. Other elements that were detected in the precipitate except Fe are mainly Mn and Mo. Particles containing Ti were observed occasionally.

4. CONCLUSIONS

Microstructure of the steel in as-delivered state is predominantly bainitic with carbide $M_3C$ ($M_2C$).

In the temperature range of 850 °C - 1250 °C significant austenite grain coarsening is evident above temperature of 1100 °C.

Recrystallization evaluation showed that the austenitization temperature of 1200 °C and deformation 65 % at temperatures of 850 °C and 1100 °C lead in all cases to significant grain refinement. During increased holding time at deformation temperature austenitic grain slightly coarsens. At regions of dead zone, where the deformation reaches (7 – 10) % (close to critical deformation value), the austenitic grain is significantly (3 - 4 times) coarser. In case of the small deformation, the dangerous grain coarsening may take effect.

After austenitization at temperature of 1200 °C and consequent deformation at temperature of 1100 °C, 70 % of volume was dynamically, respectively metadynamically recrystallized. The rest of softening proceeds by static recrystallization. In the area of possible critical deformation grain size increases approximately 3 times, but even after 5 minutes dwell at deformation temperature the grain size does not reach the size before deformation.

Deformation at 850 °C after austenitizing at temperature of 1200 °C leads to a dynamic, respectively metadynamic recrystallization only in 4 % of volume. After the dwell of 5 min at deformation temperature 80 % of volume is recrystallized.

Transmission electron microscopy showed that microstructure in all states contains particles of type $M_2C$ and $M_3C$, where M is represented by the elements Fe, Mn and Mo. Occasionally, particles of AlN and Ti(C, N) were observed. The particles are located mainly at the boundaries of austenite grains and between some martensite needles. The number of particles decreases significantly with increasing processing temperature. The particle size decreases slightly with temperature.

At present, experimental work continues with the newly supplied experimental material. The completion of the experimental program and a detailed analysis of the minority phases in a range of forming temperatures, will allow conclusions about the influence of deformation conditions on the final austenite grain size, which significantly affects the secondary structure and mechanical properties. Interest will be focused on complete evaluation of the kinetics of recrystallization, interaction of recrystallization and precipitation and assessment of conditions on the grain boundaries. Structural characteristics will be correlated with the mechanical properties of the final state of evaluated steel.

ACKNOWLEDGEMENTS

The problem was solved using laboratories of Innovation Centre for Diagnostics and Application of Materials at CTU in Prague as part of the project of the Ministry of Industry and Trade TI2/132.

LITERATURE