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Research Paper

Pinning effect of strain induced Nb(C,N) on case hardening steel under warm forging conditions

P. Springer*, U. Prahl

IEHK Steel institute RWTH Aachen University, Germany

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ABSTRACT

The effectiveness of Nb in retarding dynamic recrystallization (DRX) during warm forging process (700˚C–900 ˚C) and its dependence on the process parameters$(\varepsilon, \dot{\varepsilon}, T)$, which are the definers of formation conditions and morphology of the strain induced Nb(C,N), were studied. Due to its capacity of interaction with the austenite phase through solute drag and precipitate pinning effects, Nb is an important variable in the production of high-strength low-alloy steel (HSLA). However, the effectiveness of Nb as austenite conditioner is strictly dependent on its correct manipulation within each manufacturing step of finished or semi-finished products. Computer simulation of the precipitation kinetics of a forging steel 0.03 wt. pct. Nb was performed in order to design a thermomechanical processing route based on the time-temperature-precipitation diagram (TTP), thereby ensuring high precipitate phase fraction in short processing time. Characterization of the steel mechanical behavior and its dependence with the formation of Nb(C,N) was done through flow stress curves analysis, the relaxation rate for the microalloyed steel is remarkably lower than for the Nb free steel being a typical stress plateau (carbonitride formation) and a peak stress (recrystallization) respectively observed. Transmission electron microscopy (TEM) was performed in order to characterize the distribution, geometry and dimension of Nb(C,N) generated by different processing routes at different process stages. The solubilization capacity of the adopted and computational designed austenitizing process was also experimentally analyzed. The energy-dispersive X-ray spectroscopy (EDX) method was used in the characterization of the precipitates chemical composition, allowing the identification of Ti-rich precipitation remnants of the austenitizing process and the absence of the element in the Nb(C,N) formed during deformation (strain induced precipitates). The strain, strain rate, temperature and isothermal holding time contribution upon the formation of precipitates were evaluated separately which reveals that it is so important as the processing temperature, the strain rate regime play decisive role in the formation of the particles. In addition, the strain rate dependency of strain induced Nb-precipitates formation at optimal processing temperature was indirectly confirmed by the capacity of the formed precipitates in hinder the static recrystallization (SRX) progress under relaxation tests.

The strain induced precipitation formed at optimal processing conditions have circular geometry and size below 10 nm, causing high precipitate pinning effect observed in the conditioning of austenite phase by hindering the DRX under warm forging condition.

1. Introduction

In order to obtain refined microstructure in steel parts processed at high temperatures, it is imperative to control the process of austenite dynamic recrystallization (DRX) that can lead to a high number of nucleation sites for new phases by increasing the grain boundary fraction and/or the number of high energetic structures such as shear bands. The influence of both the mentioned microstructural features in the kinetics of ferrite phase formation as well as in its final grain size was reported in the literature by numerous authors.

Hurley et al. (2001) studied the generation of ultrafine ferrite nucleation sites in austenite during a single-pass strip rolling, it was observed that the high thermodynamic driving force generated by fast cooling combined with the shear band formation are essential to refine the grain size and at same time increase the ferrite volume fraction. Furthermore, Hickson et al. (2002) advocated the importance of shear strain modulus inherent to the rolling process, in the acceleration of ultrafine equiaxed ferrite (UFF) formation by performing different metalworking tests.

Bae et al. (2004) experimentally evidenced the effect of multiple

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* Corresponding author.
E-mail address: phillipspringer_sp@hotmail.com (P. Springer).

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Table 1
Chemical composition of steels 18CrNiMo7-6 (Nb) and reference steel grade in wt.%.

Steel	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Al	N	Nb	Ti
Nb	0.17	0.20	0.54	0.009	0.005	1.64	0.32	1.56	0.04	0.03	0.012	0.031	0.002
Ref	0.16	0.22	0.56	0.010	0.007	1.64	0.31	1.54	0.03	0.03	0.012	0.005	0.002

deformation steps in the conditioning of austenite through the control of DRX, concluding that the increase of grain effective surface area accelerates the kinetics of the austenite to ferrite transformation.

Chronologically, either by Hodgson et al. (1999) in the design of a simple thermomechanical controlled process (TMCP) to improve the mechanical properties of strips, or by Niikura et al. (2001) that in a Japanese national cooperation proposed different TMCP in obtaining ultrafine ferrite phase, or by Pan et al. (2003) that obtained ultrafine ferrite grains in different microalloyed steels, the refinement of the ferrite is highlighted as the only way to improve strength and toughness at same time in low carbon steels.

The conditioning of the austenite also plays important roles in the final strength of phases formed by diffusionless transformation. As reviewed by Krauss (1999), small prior-austenite grain size (PAGS) results in small martensite packets and consequently high strength values. The same martensite strength dependence with the PAGS was later experimentally verified by Morito et al. (2006) that highlighted the importance of the blocks, an internal subdivision of the martensite packets, in the strengthening of steel and its dependence on the chemical composition.

In this scenario the pinning effect of Nb(C,N) in the control of austenite DRX occurrence has fundamental technological importance in steel bulk forming and is also desirable in the control of austenite grain growth in heat treatment processes according to Palmiere et al. (1994).

The capacity of pinning grain boundaries by strain induced Nb(C,N) depends on the formation of a high phase fraction of fine precipitates during the process, which itself is associated with the amount of Nb in solution before start of deformation as reported by Hansen et al. (1980). In a modern TMCP the state of Nb is manipulated according to the purpose of each step. Maki (2007) showed that in such TMCP it is advantageous to have Nb either in solid solution or in Nb(C,N) form, as in austenite the pinning effect of fine Nb(C,N) are usually more effective in hindering DRX as observed by Hutchinson et al. (2008) while Nb in solution is desired as a hardenability agent according to Felfer et al. (2012).

The blockage of DRX during deformation process leads to an accumulation of strain and consequently to a not negligible increasing of processing loads in industrial scale as observed at rolling by Dutta and Sellars (1986). The balance between microstructural improvement and processing cost has to be taken in count during the design of large scale industrial processes. In bulk forming processes it is well known that the deformation accelerates the kinetics of precipitation by increasing the dislocation density as seen in Valdes and Sellars (1991), but even more important that the amount of dislocation is its arrangement in a favorable 3D network where the nodes are the energetic most favorable place for precipitates formation. As proposed by Dutta et al. (1992) on the classic theory of strain induced precipitation.

Despite of the broad acceptance of the classical theory for strain induced precipitate formation there are not many works about the influence of thermomechanical variables as strain, strain rate and temperature in the formation of such favorable dislocation configuration for the nucleation. The current work is a first author attempt to address a comparative importance to the thermomechanical parameters present in almost all bulk forming processes with special complexity of distribution in close die forging commonly solved by FE-simulation.

The characterization of the strain induced precipitates formation during warm deformation is critical due to its dependence with initial austenitic grain size, steel composition and prior amount of Nb in solution, it is necessary to apply a careful experimental method in understanding the process. The resolution of the electron microscopy performed in the present work does not discern the dislocation scale, but a consistent cause and effect analysis based on previous discussions was carried out to correlate the observed effects of various processing variables (ε, $\dot{\varepsilon}$, T) in the final precipitates configuration with the expected dislocation network.

2. Materials and methods

The investigation focuses on the Nb microalloyed case-hardening steel 18CrNiMo7-6 and uses as reference the same grade steel without Nb (Table 1). The application of 18CrNiMo7-6 steel is concentrated in the production of axles, gears and parts that after forging require surfaces hardened by thermal treatment.

The current study has as pilot process the forging of an automotive blank gear to define the processing parameter ranges of interest. The temperature is considered constant along the piece once the process takes just 2.2 s and for simplification the adiabatic heating effects are despised.

The maximum true strain applied during compression test was 1 due to the test limitation in produce reliable stress-strain data for higher values of strain, anyway it does not hinder the evaluation of the precipitates pinning effect on DRX of austenite since the DRX-onset (ε_c) for the used steel was characterized at Springer and Prahl (2016) and ranges from 0.15 to 0.18 for low strain rate regimes at warm forging condition.

The strain rate range explored in the experiments was defined by FE-simulation of an industrial one step forging process with press velocity of 20 mm/s. The temperatures of 1200, 900, 800 and 700 °C were tested during simulation but no significant difference were produced in the absolute values and distributions of strain rate throughout the gear. In Fig. 1 it is shown the simulation results produced at 800 °C, the average temperature of the defined warm forging range.

It was noted that despite the short duration time of the one-step forging process the strain rate values range from 0 to 1.2 s^{-1} through most of gear bulk volume (Fig. 1b), reaching values of 35 s^{-1} at the gear extremes (Fig. 1a). Due to the high difference of strain rate absolute value, the simulation result view was divided into two to provide a better visualization of the strain rate distribution produced in the two gear regions (Fig. 1a and 1b).

Cylindrical samples (5 mm diameter and 10 mm length) were machined to perform compression tests in a dilatometer Bähr DIL 805 Thermoanalyse with strain rates of 0.001 and 1 s^{-1}, so ensuring a reasonable approximation to the extreme cases observed in the FE-simulation (Fig. 1).

The samples were heated from room temperature at 200 °C/min up to the austenitizing temperature, austenitized for 10 min at 1250 °C, cooled at 200 °C/min up to deformation temperature (900 °C) and directly cooled at 9000 °C/min after compression or isothermal holding (Fig. 2).

In order to guarantee the constant low strain rate of 0.001 s^{-1} the compression test had to be performed at low press velocity due to the simplistic geometry and reduced length of compression samples. Coupled with the low press velocity, long times of isothermal holding (1025 s) are undergone by the steel and have to be considered in the analyzation of the precipitates found in such conditions.

To address the effect of the isothermal holding time, strain and