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# The recrystallization model and microstructure prediction of alloy 690 during hot deformation



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#### A R T I C L E I N F O

### ABSTRACT

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Keywords: Nickel-base superalloy Dynamic recrystallization Meta-dynamic recrystallization Grain growth Finite element method The recrystallization model and microstructure evolution during hot deformation of alloy 690 are investigated by the hot compression tests in a temperature range of 1000–1250 °C and at strain rates of  $0.01-10 \text{ s}^{-1}$ . A series of integrated microstructure prediction models for alloy 690 including dynamic recrystallization (DRX), meta-dynamic recrystallization (MDRX) and grain growth are developed in consideration of the actual requirement of hot deformation simulation. The accuracy of the models is validated using the finite element method (FEM) by comparing the simulation result with real manufactured one. Furthermore, the FORTRAN language is used to carry out secondary development of DEFORM-2D for the effectively invoking of the developed models in the hot extrusion simulation. The simulated microstructure agrees well with the microstructure of alloy 690 pipe obtained from the actual hot extrusion process in the factory, indicating the model developed in present study can be used for theoretical guidance in hot extrusion.

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#### 1. Introduction

Alloy 690 is an austenitic nickel-base alloy, which is proposed as a substitute material for Alloy 600 due to its much higher chromium concentration (28-31 wt% vs 15-17 wt%) [1] and superior resistance to stress corrosion cracking (SCC) [2]. The manufacture process of nickel-base alloy pipe involves a series of procedures including homogenization of the ingot, hot extrusion, cold rolling of the tube and heat treatment [3]. The hot extrusion is a crucial step in the manufacture process, because not only cracks of the pipe may occur, but also the microstructure can be adjusted by hot working process. The desired mechanical and microstructure properties of nickel-base alloy after hot deformation are strongly depend on the deformation parameters, such as temperature and strain rate [4]. Furthermore, due to the fact that the superior resistance to SCC and other excellent properties of alloy 690 are closely related to the grain size and microstructure uniformity [5], obtaining a uniform microstructure by hot working process makes great sense for alloy 690 [6,7]. However, previous study shows that the high content of alloying elements makes the microstructure control during hot working difficult for nickel-base alloy [8]. Though there have been a large number of investigations on alloy 690, most of them focus on the corrosion resistance [9], fatigue properties [10], precipitation behavior [11] and heat treatment [1,12]. In the limited investigations on hot deformation of alloy 690, Guo et al. established the processing map of alloy 690 [7] and Wang et al. investigated the

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dynamic recrystallization (DRX) mechanism of the alloy [13], however, no work has been reported on the effort for microstructure control and optimization during hot deformation of alloy 690.

In addition, the variation of grain size is significantly related with DRX, meta-dynamic recrystallization (MDRX) and grain growth during hot deformation [14]. Thus, the hot extrusion process is a crucial step to get alloy 690 pipes with desirable microstructure. As to this, it is necessary to investigate the hot deformation process and manufacture parameters optimization of alloy 690. The finite element method (FEM) has been proved to be a powerful and accurate microstructure prediction method in the investigation of hot deformation process [15–18]. The application of FEM in material forming has brought great changes to avoid the traditional trial and error approaches [19], which can be employed in the microstructure prediction and design for alloy 690 pipes. It is noteworthy that the accuracy of the kinetics models is essential for any precise finite element simulation [20]. The importance of accurate DRX model in the microstructure prediction during hot deformation simulation has been proved by many reports [20,21]. Meanwhile, DRX and related microstructure evolution occur during hot deformation, leaving the microstructure in an unstable state which provides the driving force for MDRX and grain growth after hot deformation [22]. As a result, the MDRX and grain growth models also have to be taken into consideration for accurate microstructure prediction by FEM. Though the DRX kinetic model of alloy 690 has been reported in some papers [23,24], it is not sufficient for the accurate microstructure prediction by FEM. As to the lack of database for alloy 690, there is an urgent need for the study of an integrated microstructure evolution model including DRX, MDRX and grain growth process,

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which shows great importance in the microstructure control and deformation parameter optimization during manufacture process of alloy 690 pipes.

Based on the above facts and motivations, the models of DRX, MDRX and grain growth for alloy 690 are investigated and established based on the data derived from the Gleeble-1500 thermo-mechanical simulator. The FEM is used to validate the accuracy of the established model by comparing the simulation results with the experimental ones. This model is then used for the predict of microstructure evolution during hot extrusion of alloy 690 in actual manufacture process. Different from previous studies, a series of integrated microstructure evolution models involved in hot deformation are developed and the models are validated by the DEFORM-2D software.

#### 2. Materials and experimental procedure

#### 2.1. Materials

The experimental material of alloy 690 used in this study is produced by vacuum induction melting (VIM) and electroslag remelting (ESR) processes, followed by homogenization and cogging. The chemical composition is shown in Table 1. Different solution treatments were performed on the materials to get specimen with grain size of 35, 125 and 211 µm.

#### 2.2. Hot compression tests

A series of hot simulation compression tests were carried out on a Gleeble-1500 thermo-mechanical simulator to investigate the DRX, MDRX and grain growth behavior of alloy 690. Cylindrical compression specimens for hot compression are 8 mm in diameter and 12 mm in height. During the hot compression process, a mica sheet was used as lubricant between specimen and compression dies in order to ensure the uniform of deformation. The true stress–strain data was recorded by the controlling computer equipped with an automatic data acquisition system.

#### 2.2.1. Hot compression for DRX model

The DRX behavior is critical for microstructure control during hot deformation. In order to establish the DRX model of alloy 690, the single pass compression experiment was conducted for the samples with an initial grain size of 125  $\mu$ m in the temperature range of 1000–1250 °C with a strain rate of 0.01–10 s<sup>-1</sup> to a true strain of 0.16–0.9. The specimens were heated to the deformation temperature with a rate of 20 °C/s and holding for 30 s for the temperature uniform and then compressed to the given true strain as illustrated in Fig. 1.

#### 2.2.2. Hot compression for MDRX model

Double pass compression experiments were conducted for the samples with 3 different grain sizes to detect the MDRX behavior of alloy 690. Fig. 2 is the schematic diagram for the double pass hot compression. Specimens were compressed at deformation temperature of 1100–1200 °C with strain rate of  $0.1-10 \text{ s}^{-1}$  and the interval time  $\Delta t_1$  varying from 0.5 s to 15 s as shown in Fig. 2(a). Both passes were deformed to a true strain of 0.16 for the determination of MDRX kinetic model of alloy 690. In order to establish the MDRX grain size model, the single pass compression experiment is conducted for the samples with 3 different grain sizes of 35 µm, 125 µm and 211 µm compressed with the same deformation parameters in Fig. 2(a) and then held for a fully MDRX time  $\Delta t_2$ . The fully MDRX time  $\Delta t_2$  is determined by the

#### Table 1

Chemical components of alloy 690 used in the experiment (wt%).

Cr	Fe	С	Ti	Al	Ni
29.19	9.49	0.013	0.24	0.28	Bal



Fig. 1. Schematic diagram of single pass hot compression tests for alloy 690 to investigate DRX.

calculation of MDRX kinetic model. Fig. 2(b) illustrates the single pass compression for the establishment of MDRX grain size model.

#### 2.2.3. Hot compression for grain growth model

Grain growth will also take place in the manufacture process by hot deformation. The establishment of grain growth model is based on one pass hot compression tests as illustrated in Fig. 3.

All the samples were water-cooled immediately after hot compression in order to keep the deformation microstructure. The deformed samples were cut through in the direction parallel with the compression direction. The cut surfaces were mechanically gridded to 2000 # and polished with 2.5  $\mu$ m polishing paste, then boiled in the solution of 10 ml H<sub>2</sub>SO<sub>4</sub> + 2 g KMnO<sub>4</sub> + 90 ml H<sub>2</sub>O for 30–40 min for optical microscope (OM) observation. The central part of the sample was used for the observation. The average grain size was calculated by the line intercept method.

#### 2.3. Finite element simulation and verification

The finite element simulation was carried out by DEFORM-2D commercial software to validate the accuracy of the established model and predict microstructure evolution during hot extrusion. Although DEFORM-2D software can conduct analysis and calculation of macroscopic characteristics, it does not have perfect simulation and prediction ability of microstructure evolution. Thus FORTRAN language was used for secondary development of DEFORM-2D software and the hot deformation microstructure evolution models of alloy 690 were compiled into the user defined subroutine to analyze the feasibility of microstructure of alloy 690 pipe was analyzed to compare with the prediction by DEFORM-2D.

#### 3. Results and discussion

#### 3.1. The dynamic recrystallization model

#### 3.1.1. The flow behavior

The flow behavior of alloy 690 under different deformation conditions are demonstrated in Fig. 4. The flow curves are characterized by a rapid increase of stress to a peak value followed by a gradual decrease or steady state. In the work hardening stage, the dislocations increase and accumulate rapidly, causing obvious work hardening [17]. When the accumulated dislocation density reaches a critical value, DRX takes Download English Version:

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