Study on Microstructure of directional solidified T91 steel

Q S Wang¹, W Q Wang¹* and Z M Shi²

¹College of Materials Science and Engineering, Jilin University, No.5988 Renmin Street, Changchun 130022, China
²Zhejiang Industry & Trade Vocational College, No.717 Fudong Street, Wenzhou 325002, China

*E-mail: wwq@jlu.edu.cn

Abstract. Using the method of directional solidification to prepare T91 steel is an effective way to reduce the elastic modulus and improve the thermal fatigue properties of alloy. In this paper, T91 steels were fabricated through liquid metal cooling method by the solidification rate of 30μm/s, 90μm/s and 180μm/s, and the crystal morphology was analysed. The results demonstrate that deflection angle of columnar crystals is increased with the raising of solidification rate, the maximum angle is 20 degrees, and the solidification structure is refined in general. The central solidification zone is the concentration area of directional microstructures, in which lath ferrites are arranged in parallel and the martensites are mainly distributed in the beginning and the end of the sample. The range of elastic modulus values of three specimens is 141.05~186.73GPa, and E¹80μm/s<E¹30μm/s<E¹90μm/s<E¹common cast. The thermal fatigue property of T91 obtained by directional solidification method is better than that by common casting. When the solidification rate is 90μm/s, the thermal fatigue resistance of the specimen is best, which increases by about 25%.

1. Introduction

T91 steel is a typical ferritic/martensitic heat-resistant steel [1]. It has high thermal strength, low production cost, and also has good ductility, oxidation resistance and corrosion resistance, so it is widely used in Superheater, reheater and heat resistant pipe of power station [2, 3]. At present, the researches on T91 steel mainly focus on the stress analysis and finite element modelling of dissimilar joints [4]. For instance, Mittal R used nickel base welding wire to weld T91 steel and 347H steel by TIG welding, which obtained the joints with higher tensile strength and better plasticity [5]. Gao Wei used the finite element software to simulate the residual stress distribution of the T91/12Cr1MoV welded pipe [6]. However, most of these researches are aimed to improve the plasticity, strength and other mechanical property of ferritic heat-resistant steel, the research on improving the thermal fatigue property of T91 steel is very few.

Thermal fatigue is caused by the temperature gradient changed by the cycle, and has a direct relationship with the elastic modulus of the material itself [7]. Data shows that, the columnar crystals arranged in a fixed direction can obviously reduce the elastic modulus of materials [8]. And directional solidification can obtain columnar crystals with specific orientation by setting up a temperature gradient along a specific direction in the solidified and uncured melts, so it is an effective way to reduce the elastic modulus, improve the thermal fatigue property and high temperature creep performance of alloy [9].
2. Experimental methods
The chemical composition of T91 steel was listed in table 1.

| Element | C    | Cr  | Mo  | Si  | V   | Mn   |
|---------|------|-----|-----|-----|-----|------|
| Content | 0.08–0.12 | 8.00–9.50 | 0.85–1.05 | 0.20–0.50 | 0.18–0.25 | 0.30–0.60 |

| Element | Nb   | N   | P   | S   | Al  | Ni   |
|---------|------|-----|-----|-----|-----|------|
| Content | 0.06–0.10 | 0.03–0.07 | ≤0.020 | ≤0.010 | ≤0.040 | ≤0.040 |

The directional solidification device included feeding system, vacuum system, liquid metal cooling system and pumping system. The experimental process was: firstly, put T91 steel into the ceramic membrane, heated Sn to liquid until the experiment was over. Pumped the furnace to vacuum, rose the temperature to 1580℃ for 30min. Then pulled the ceramic membrane down to Sn liquid with the speed of 30μm/s, 90μm/s and 180μm/s. When the sample was completely solidified, got it out.

Cut the samples like figure 1, then stuck strain gauges on them, kept accounts of strain and stress value during the tensile test. The tensile rate was 10^{-5}/s, maximum strain was 0.2%. Fitted stress-strain curve and calculated the elastic modulus.

![Figure 1. The shape and size of tensile specimen.](image)

3. Results and discussion

3.1. Observation and analysis of T91 surface
Figure 2 is the macroscopic morphology of the solidified sample after corrosion. The solidification rates are 30μm/s, 90μm/s and 180μm/s, they are respectively labelled sample a, sample b, sample c. As can be seen, a large number of columnar crystals and dendrites form the structural state of the whole sample. The direction of white markings in the figure is the extension direction of the solidified tissue. Sample a, for example, while the main heat flux is flowing downward, part of heat flux moves to the left, about 5°. Similarly, the grain direction on the right is deflected 5 degrees to the right. The formation direction of sample b’s grain is approximately same to sample a, but the surface is good, and defect is less. Sample c has rough surface, A few shrinkage holes and small cracks appear, this is because the moving speed is too high, and cooling process of the sample is fast, solidified liquid metal can’t be filled in time.
3.2. The phase analysis of solidified tissue

In order to obtain excellent solidification structure of T91 steel, the orientation of dendrites should be ensured first, and the thermal fatigue’s direction should be consistent with the direction of liquid metal. Figure 3 is the morphology of the directional tissue obtained by joining the metallographic photos of each position. The grain size of sample b is most regular, and the grains are almost parallel to the crystal growth direction. The grain structure is mainly composed of lath ferrite, delta ferrite, austenite and longitudinal martensite. The edges of the lath ferrites are relatively straight, they gather in the middle of sample and the delta ferrite is arranged like a short rod along the direction of growth. The width of grain is 200~380μm.

On the basis of ensuring the single orientation of ingot structure, the grain size should be refined as much as possible, because more slip planes can be carried out, decrease the deformation and probability of crack initiation. The method of statistical grain size is: make a straight line along the width direction of the steady growth zone (as shown in figure 3), the more the grain number on the line, the smaller the solidification structure is. After four measurements, the average value is shown in table 2. The grain number of sample a, b and c is 15, 24 and 26. Therefore, the solidification rate of sample b and c is better relatively.
Table 2. Grain numbers in different positions.

| Sample | Grain number in different positions | Average value |
|--------|-------------------------------------|---------------|
| a      | 14 18 16 12 15                      |               |
| b      | 28 19 26 23 24                      |               |
| c      | 21 20 34 29 26                      |               |

Figure 4 is the XRD test result of directionally solidified sample. It is known that the phase of T91 steel is mainly NbC, VN and Cr₂₃C₆. Cr₂₃C₆ is a M₂₃C₆ type interstitial compound (M stands for metallic elements), it mainly exists on martensite lath, the nucleation site is located at austenite and martensite grain boundaries. Both NbC and VN phases belong to MX type compounds (X stands for nonmetallic elements), they are mainly located in martensite, the size is smaller than Cr₂₃C₆. MX type compounds form nucleus in the martensitic dislocations, retaining a coherent lattice relationship with martensite. Nb and V are strong carbides forming elements, which are easy to combine with C and N, so the dispersion strengthening of NbC and VN is very good, and it’s not easy for carbides to transfer and dissolve. Macroscopically, the thermal stability of T91 steel has been improved.

![XRD pattern of T91 steel](image)

Figure 4. The XRD pattern of T91 steel.

Figure 5 is the microstructure of sample c under SEM. Figure 5 (a) is taken from the center position of the sample (stable growth area), the main phase of the region is slate ferrite, grain size is fine and tidy. Figure 5 (b) is near the side of the sample. The left side of the picture is the sample’s center. As shown in the picture, regular columnar crystals is formed by the influence of directional solidification heat flow. Besides, under the effect of transverse heat flow, the direction of the right side grain changes a lot, and there is a clear dividing line with the left side, the angle is about 45°. This shows that under the influence of several temperature gradients, the grain direction of the ingot edge begin to dissimilate. However, in the central region, the direction of the heat flow is single and straight, that means the directionality of the ingot center’s microstructure is better.
3.3. Analysis of elastic modulus and fatigue properties

The fatigue of metals is closely related to the plastic strain, and the relationship of them is $N_f = K(\Delta\varepsilon_p)^{-c}$, $N_f$ is low-cycle fatigue plastic strain, $\Delta\varepsilon_p$ is plastic strain, $K$ and $c$ are constants, the total strain of the material is affected by both elastic strain and plastic strain: $\Delta\varepsilon_p = \Delta\varepsilon_e + \Delta\varepsilon_p$. When the total load is constant, the elastic strain should be increased if the plastic strain is reduced. Table 3 is the elastic modulus at different locations measured by tensile test. It can be seen that the elastic modulus of ordinary casting is 185.97 GPa, and $E_{90\mu m/s} < E_{180\mu m/s} < E_{30\mu m/s} < E_{\text{common cast}}$. Consequently, when the solidification rate is 90μm/s, the thermal fatigue property of the sample is the best, which is 25% higher than that of the common casting.

Table 3. The elastic modulus of four samples at different positions (GPa).

| Positions | Common casting | Sample a | Sample b | Sample c |
|-----------|----------------|----------|----------|----------|
| 1         | 184.2          | 186.73   | 169.75   | 173.69   |
| 2         | 187.1          | 164.68   | 178.26   | 168.17   |
| 3         | 187.5          | 168.28   | 142.67   | 165.40   |
| 4         | 183.6          | 171.32   | 141.05   | 173.21   |
| 5         | 188.2          | 170.56   | 159.14   | 165.93   |
| 6         | 185.2          | 178.26   | 166.67   | 171.24   |

4. Conclusions

(1) By the method of directional solidification, samples with orientation growth grains were obtained at three solidification rates of 30μm/s, 90μm/s and 180μm/s. With the increase of solidification rate, the overall deflection angle of columnar crystals increases and the microstructure is refined. When the solidification rate is 90μm/s, the surface of the sample is smooth and the grain continuity is the best.

(2) The initial solidification zone of T91 steel is mainly martensite, it is mixed up with white $\alpha$-Fe and some small retained austenite. A large number of parallel arranged lath ferrite appeared in the stable growth zone, the width of grain is 200~380μm.

(3) The range of elastic modulus of three directional solidification specimens is 141.05~186.73 GPa, and $E_{90\mu m/s} < E_{180\mu m/s} < E_{30\mu m/s} < E_{\text{common cast}}$. The elastic modulus of T91 steel obtained by directional solidification is lower than that of common casting, the thermal fatigue properties improves obviously. When the solidification rate is 90μm/s, the thermal fatigue resistance is improved by about 25%.

References

[1] Gigax J G, Chen T and Kim H Radiation response of alloy T91 at damage levels up to 1000 peak dpa 2016 *J. Nucl. Mater* **482** 257
[2] Song M, Sun C and Fan Z A roadmap for tailoring the strength and ductility of ferritic/martensitic T91 steel via thermo-mechanical treatment 2016 Acta. Mater 112 361
[3] Wang W Z, Wang Y and Zhu Y M Research status and advances of manufacture and properties of P91/T91 steel in China 2010 Mat. Mech. Eng 34 6
[4] Haney E M, Dalle F and Sauzay M Macromolecular results of long-term creep on a modified 9Cr–1Mo steel (T91) 2009 Mat. Sci. E A 510 99
[5] Mittal R and Sidhu B S Microstructures and mechanical properties of dissimilar T91/347H steel weldments 2015 J Mater. Process.Tech 220 76
[6] Gao W, Jiang Y and Gong J M Numerical simulation and analysis of welding residual stress in T91/12Cr1MoV dissimilar welded joint 2012 P. CSEE 32 126
[7] Li Z Y, Liu L J and Nan X H Role of marangoni tension effects on the melt convection in directional solidification process for multi-crystalline silicon ingots 2012 J. Cryst. Growth 346 40
[8] Delaleau P, Beckermann C and Mathiesen R H Mesoscopic simulation of dendritic growth observed in X-ray video microscopy during directional solidification of Al-Cu alloys 2010 Isij International 50 1886
[9] Brundidge C L, Miller J D and Pollock T M Development of dendritic structure in the liquid-metal-cooled, directional-solidification process 2011 Metall Mater Trans A 42 2723