

Freckles pattern and microstructure feature of Nb-Ti alloy produced by vacuum arc remelting

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Abstract

Nb-Ti alloys are normally produced by vacuum arc remelting process. Due to inadequate processing parameters, freckles can be observed in macroetched ingots. In the present work, visual, chemical, metallographic, and X-ray are presented which establish the appearance, composition, microstructure differences between freckle regions and normal regions. It has been observed that in freckles parallel to the ingot axis, the Ti content is up to 53wt%, 7wt% higher than normal regions. It is also shown that a lot of precipitation phases appeared in freckle regions, because of the thermosolutal convection in the mushy zone. The Rayleigh number, which recommended as a criterion for freckle initiation, has been calculated that using a VAR melting software. Based on the experimental results and simulation results, it is concluded that freckles can be influenced by processing parameters, and freckles in Nb-Ti alloy can be eliminated by matching appropriate processing parameters.

Keywords: Nb-Ti alloy, Freckle, Vacuum arc remelting

1. Introduction

The most widely used superconducting materials are based on Nb-Ti alloys with Ti contents ranging from 46-50 weight % Ti. These alloys of Nb and Ti have both high strength and ductility and can be processed to achieve high critical current densities that make them ideal candidates for magnet and applications. Nb-Ti alloy ingots are usually obtained by vacuum arc remelting (VAR), because of inadequate control of the alloy melting process can result in macrosegregation defects, such as niobium inclusions, freckles and tree-rings. High J_c requirements superconductors required high degree of chemical homogeneity, demanding no Nb inclusion and a minimal number of freckles.

Freckles, channel-like macrosegregation defects, are commonly observed in alloys with different melting points and densities of elements, such as Pb-Sn alloys, nickel-based superalloys, etc. The origin of freckles during unidirectional solidification is studied in a transparent, low melting model system, 30 weight % $\text{NH}_4\text{Cl-H}_2\text{O}$ [1]. In the investigation, S M Copley et al. have directly observed the formation of freckle trails in a unidirectionally solidified transparent model system. On the basis of

these observations, a mechanism for freckling is proposed. J R Sarazin et al [2]. systematically studied the freckles in Pb-Sn alloys, and proposed the Rayleigh number to predict the formation probability of freckles. In recent years, three-dimensional (3-D) microscale numerical model that predicts microsegregation, dendrite morphology and inter-dendritic convection is presented and applied to investigate the mechanisms of freckle initiation at the dendritic level by Y Luan et al [3]. Overall, it is generally agreed that freckles arise due to a complex interaction of solute segregation, thermal variation and dendrite morphology, all of which contribute to the onset of thermosolutal convection in the mushy zone [4-10].

In this paper, the freckles pattern and microstructure of Nb-Ti alloy were studied.

2. Experimental method

A 500-mm-diameter ingot of Nb-47wt%Ti alloy produced on an industrial-scale VAR furnace was examined. The major process parameters such as melting rate, current, voltage, vacuum, etc. were recorded and monitored by the furnace control system (PLC). Also, a number of whole melting processes were videotaped.

The VAR ingot was cut into three transverse sections. Each of these individual sections was cut longitudinally along the nominal ingot centerline, and then cut to produce about 15-mm-thick slices from the front faces of each of the three sections. The slices were then labeled from slice L1 (the top of the ingot) to slice L3 (the bottom of the ingot). Three semi-circular ingots, which located on the same side of the original ingot) are selected, 15-mm-thick slices are cutted 200 mm and 900 mm away from the head of the original ingot, labeled as T1 and T2.

The longitudinal slices were macroetched using 70 pct HCl and 30 pct HNO₃, to reveal the grain structure. The X-Ray scanning, a qualitative method of chemical homogeneity measurement, were used to measure macro-segregation on both the longitudinal slices and transverse slices.

The microstructural observations were made on a Olympus GX71 optical microscope using reflected light and digital imaging software. The compositional analysis (as-polished samples) was conducted on a JEOL-6460 scanning electron microscope interfaced with a Link energy dispersive spectrometry (EDS) facility.

3. Results and Discussion

3.1 Macroscopic Characteristics of VAR Ingot

Figure 1 shows macrostructure for the longitudinal slices, freckles and tree rings can be observed. Tree rings are along the outer periphery of the ingot, were a kind of macro-segregation caused by

periodic fluctuation of solidification rate in the melting process, can be used to determine the melt pool profile. Freckles, occurs only in the upper 1/3 of the ingot (L1 slice), are mostly found in the locations between the midradius and center of the ingot.

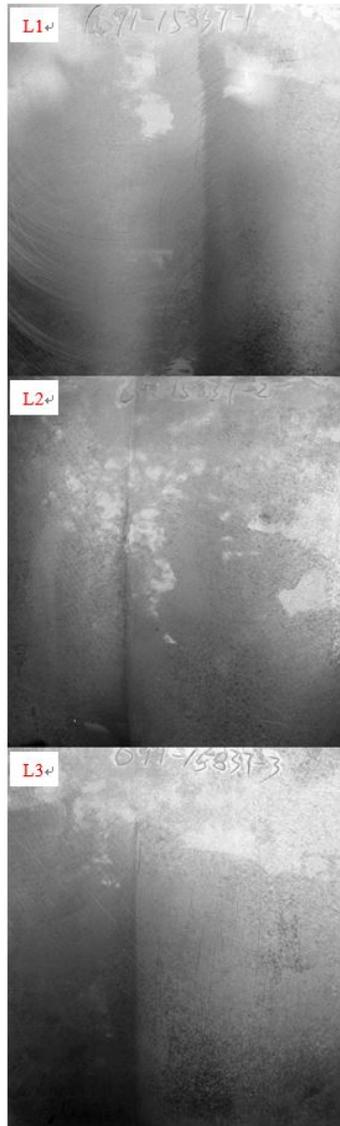


Figure 1. Longitudinal cross sections L1, L2, and L3. L1-the top of the ingot, L2-the middle of the ingot, L3- the bottom of the ingot

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Figure 2 and figure 3 show X-ray scanning flash radiograph for the longitudinal slices and the transverse slices respectively.

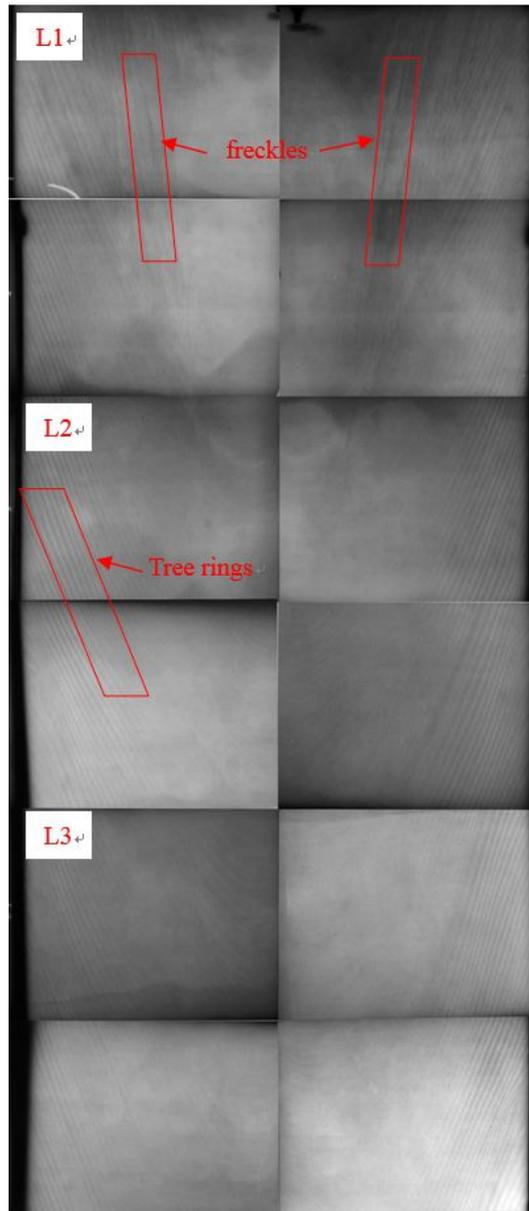


Figure 2. X-ray scanning flash radiograph for the longitudinal slices. L1-the top of the ingot, L2-the middle of the ingot, L3- the bottom of the ingot

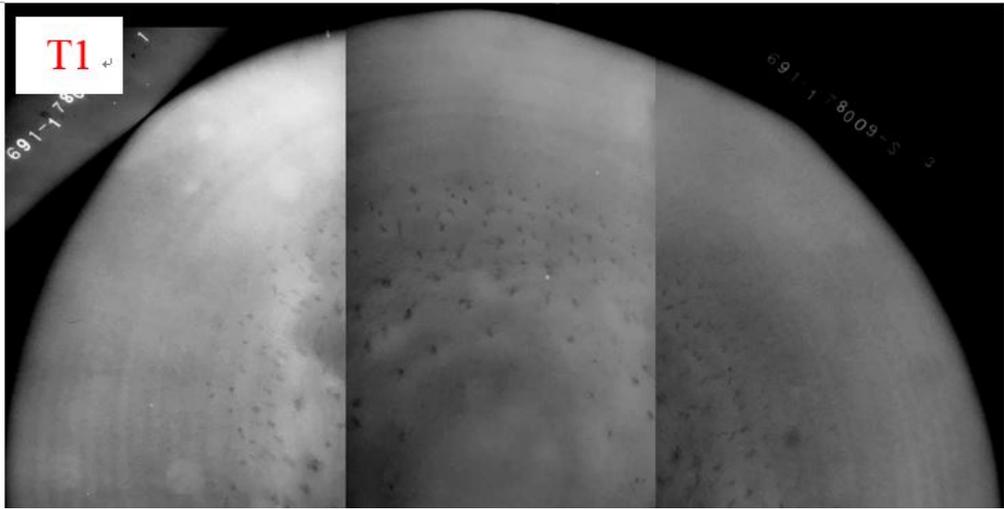


Figure 3. X-ray scanning flash radiograph for the transverse slices

Similar to macrostructure, freckles only appeared on slice L1 and slice T1. Freckles are long strips in the longitudinal direction and dots in the horizontal direction, which are dark gray. According to the principle of X-ray detection, elements with low density show dark, so it can be qualitatively determined that titanium is enriched in freckle area.

3.2 Microscopic Characteristics of Freckles

A sample at the marked position of slice L1 was sectioned, polished/etched, and examined in order to reveal detailed features and the composition of freckle defects. As shown in Figure 4, there are a large number of precipitates in the freckle region.

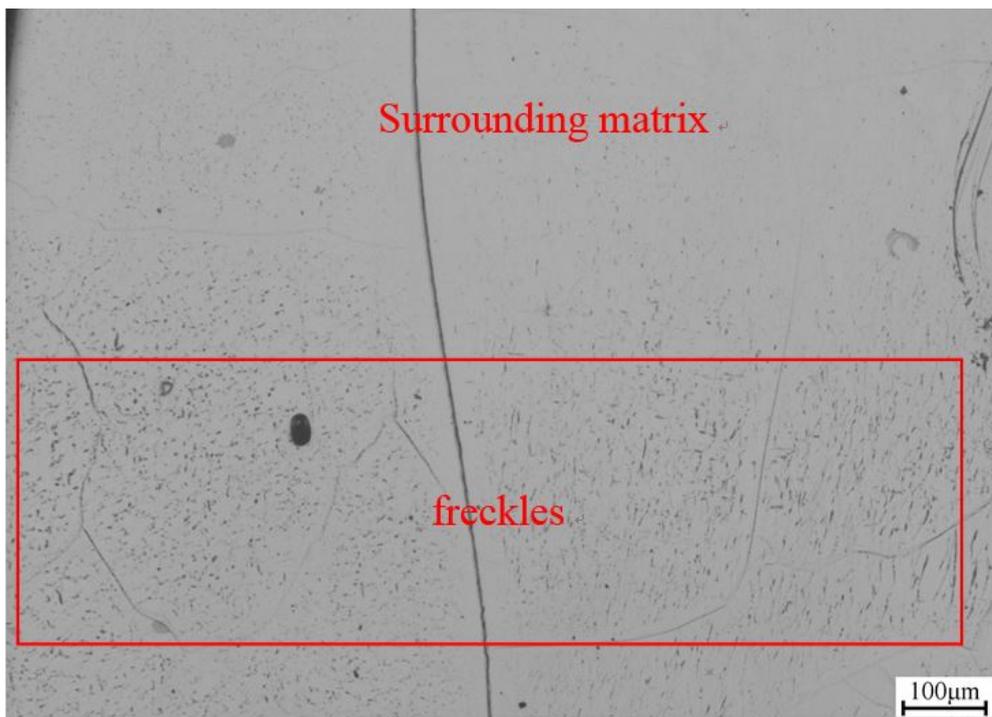


Figure 4. Optical micrographs of freckle region, sampled from slice L1

A longitudinal sample was analyzed by SEM/EDS. The compositions of freckles and their surrounding matrix are shown in Tables 1. The results are the average compositions from the measured results at least five different locations. Significant compositional difference can be detected between the freckles and the matrix. The freckles are enriched with Ti compared with the surrounding matrix, the Ti content of freckles is 7wt% higher than surrounding matrix.

Table 1. Compositions of Freckle and the Surrounding Matrix Area(Weight percent)

Element	Ti	Nb
Freckle	54.45	45.55
Matrix	47.60	52.40

In addition, precipitates in freckle area were analyzed by SEM/EDS, SEM. Results are shown in Figure 5 and EDS results are shown in Table 2. The results are the average compositions from the measured results at least five different locations. Ti

content in the precipitated phase region is higher than that in the freckle region as a whole.

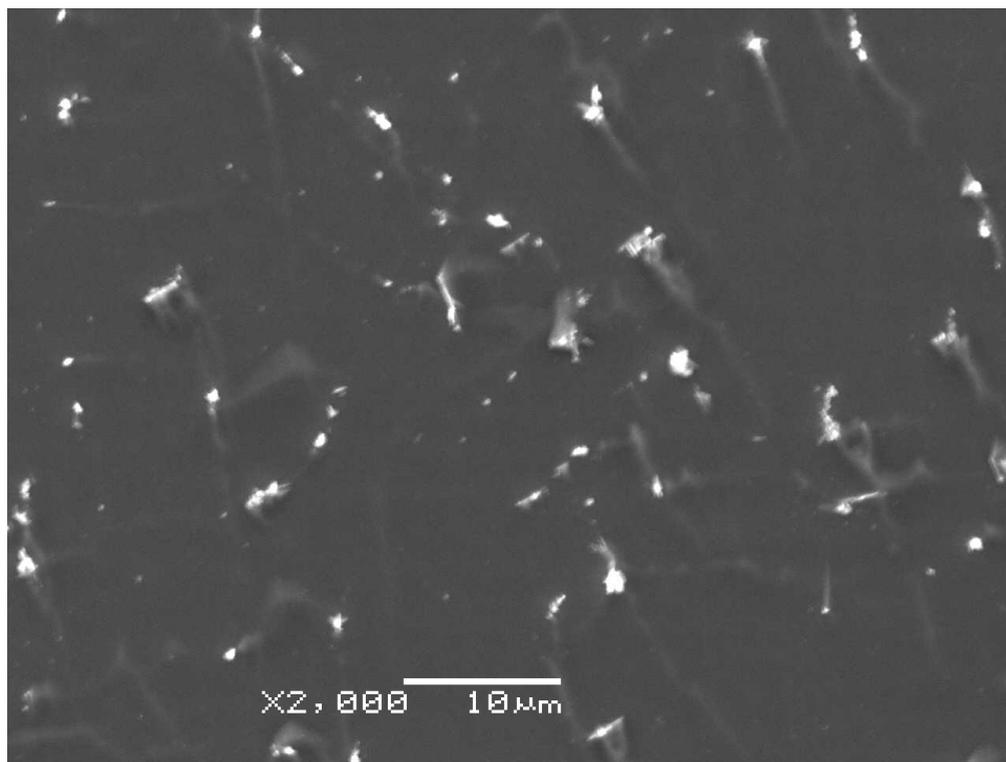


Figure 5. SEM microstructure of freckle regions, sampled from slice L1

Table 2. Compositions of precipitates in Freckle area (Weight percent)

Element	Ti	Nb
precipitates	56.96	43.04

3.2 Freckles modeling and eliminated

The Rayleigh number, a ratio proportional to the buoyancy driving force over the viscous resistance force, has been recommended as a criterion for freckle initiation [11-13]. When the Rayleigh number is less than the critical Rayleigh number, the probability of freckle formation is small. Conversely, the probability of freckle formation is high.

$$R_a = \frac{gKh}{\alpha\nu} \left(\frac{\Delta\rho}{\rho} \right)$$

where h is the characteristic length scale, g is the acceleration due to gravity, K is the mean permeability of the mushy zone, a is the thermal diffusivity, ν is the kinematic viscosity, and $\Delta\rho/\rho$ is the density inversion due to thermal and/or compositional variation.

The Rayleigh number of different processes was simulated by using Meltflow simulation software, as shown in Figure 6. According to the simulation results, the melting process was optimized, and the whole ingot without freckle was obtained. The imaging results of the transverse and longitudinal X-ray scanning flash radiograph are shown in Figure 7 and Figure 8

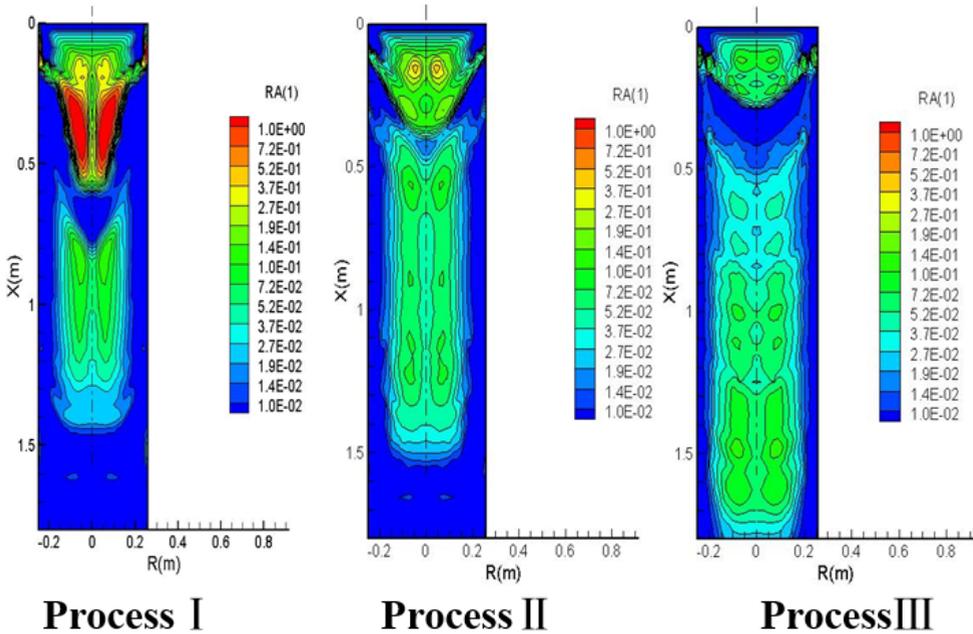


Figure 6. Rayleigh number simulation results under different processes

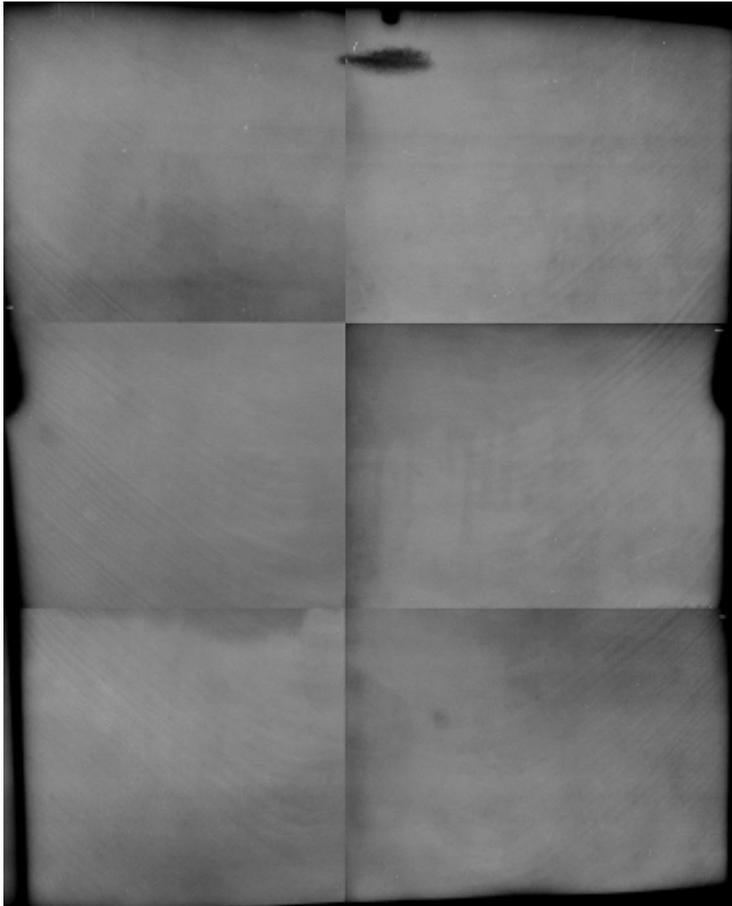


Figure 7. X-ray scanning flash radiograph for the longitudinal slice of Nb-Ti ingot prepared by optimizing process, upper 1/3 of the ingot

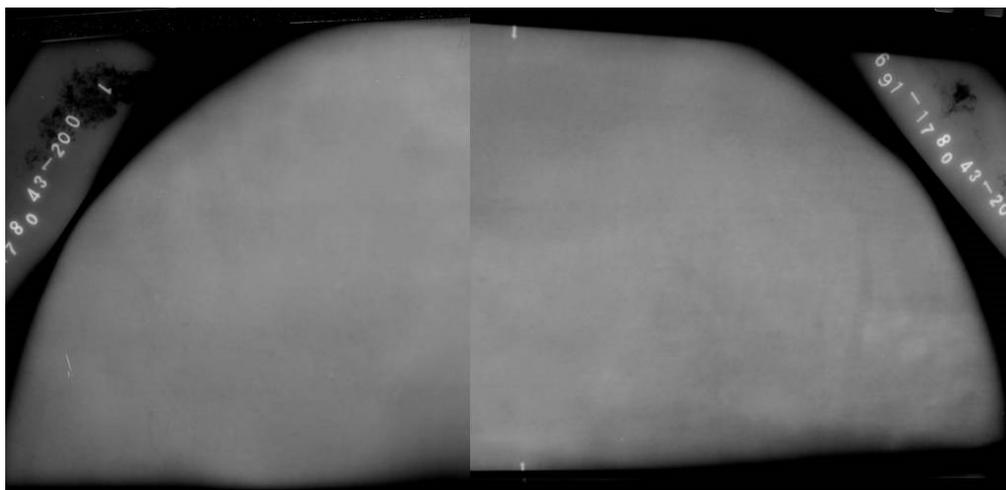


Figure 8. X-ray scanning flash radiograph for the transverse slice of Nb-Ti ingot prepared by optimizing process, 200 mm away from the head of the original ingot

4. Conclusion

In this paper, the macrostructure, microstructure and composition of freckles in Nb-Ti alloy have been studied, the results of the experimental investigation presented previously can be summarized as follows.

1. Freckles are mostly found in the locations between the midradius and center of the ingot, which is thin strip in longitudinal slice and dot in transverse slice.
2. The microstructure of freckle area was analyzed by optical microscopy and scanning electron microscopy. It was found that a large number of precipitated phases were dispersed in the freckle area. EDS analysis showed that the freckles are enriched with Ti compared with the surrounding matrix, while precipitates in freckle area were slightly higher than that in other parts of freckles.
3. The factors affecting the freckle formation probability of Nb-Ti alloy were obtained by simulation calculation. The Niobium-Titanium ingot without freckle was obtained by optimizing the melting process parameters. Through the simulation calculation, the influencing factors on the freckle forming probability of Nb-Ti alloy were obtained. By controlling these factors reasonably, Nb-Ti ingots without freckle can be obtained.

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