

PAPER • OPEN ACCESS

## Effect of the Aging Treatment on the Precipitates and Vickers Hardness of Friction Welded Super304H Joints

To cite this article: Lu Yuan *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **470** 012029

View the [article online](#) for updates and enhancements.



**IOP | ebooks™**

Bringing you innovative digital publishing with leading voices to create your essential collection of books in STEM research.

Start exploring the collection - download the first chapter of every title for free.

# Effect of the Aging Treatment on the Precipitates and Vickers Hardness of Friction Welded Super304H Joints

Lu Yuan<sup>1,a</sup>, Li Debiao<sup>1</sup>, Zhang Jianlong<sup>1,2</sup>, Shang Kang<sup>1</sup>, Yang Yang<sup>1</sup> and Cao Bo<sup>1</sup>

<sup>1</sup> Xi'an Special Equipment Inspection Institute, Xi'an 710065, China

<sup>2</sup> School of Mechanical Engineering, Xi'an University of Science and Technology, Xi'an 710054, China

<sup>a</sup> luyuan19801104@163.com

**Abstract:** In this paper, the precipitates and vickers hardness of the friction welded Super304H joints were studied after aging at 625 °C for different time. Cr<sub>23</sub>C<sub>6</sub> and NbC is denoted precipitated phases in the X-ray diffraction profiles. Precipitation, growth and aggregation of the Cr<sub>23</sub>C<sub>6</sub> carbides were the main precipitated phase evolution mechanism. The Vickers hardness of the joints increased firstly, than decreased when after aged for 500 h, this is attributed to the aggregation and growth of the M<sub>23</sub>C<sub>6</sub>.

## 1. Introduction

The increase in operating steam pressure and temperature of the conventional boiler/steam turbine power plant will result in increase in efficiency of the power plant. The increase in efficiency leads to less fuel consumption and thereby reduces CO<sub>2</sub> emission. Critical research is ongoing in the area of development of newer creep resistant, oxidation resistant and corrosion resistant materials which are capable of operating under high stresses. For these reasons, the austenitic stainless steels are the primary candidates in the superheater/reheater tubing, where oxidation resistance and fireside corrosion resistance become important in addition to the creep strength [1]. The recently developed Super304H (0.1C-18Cr-9Ni-3Cu-Nb-N) austenitic steel has shown considerable promise due to its high oxidation resistance, corrosion resistance and creep resistance. The Super304H has higher strength at elevated temperatures are required for super heater tubes in fossil fired boilers. This excellent creep rupture strength is based on finely precipitated particles such as Nb(C, N) and NbC [2,3]. Friction welding is a solid state welding process where the welding takes place well below the melting temperature of the alloy, combined with the autogenous nature of this welding process nullifies adverse effects of low temperature eutectics segregation and the uncertainties in the filler metal selection. The process is a automatic process which can produce quality welds independent of the operator's skill and can offer advantages on environmental issues. This illustrates the benefits associated with establishing a welding procedure for joining of pipes/tubes by friction welding. The friction welding can be successfully applied for welding of austenitic stainless steel joints for higher temperature application [4-6]. In order to use friction welded Super304H joints widely in higher temperature environment, the effect of aging time on the properties of the friction welded Super304H joints is investigated. This study aims to investigate the precipitates and Vickers hardness of the friction welded Super304H joints after aged at 625 °C. The analysis of precipitates and Vickers



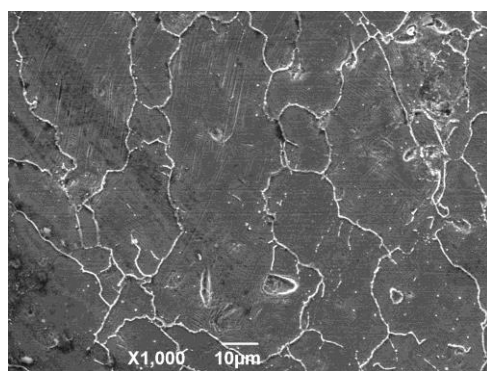
hardness at different aging time provides the more fundamentals for wide application of the friction welded Super304H joints at higher temperatures.

## 2. Experimental procedures

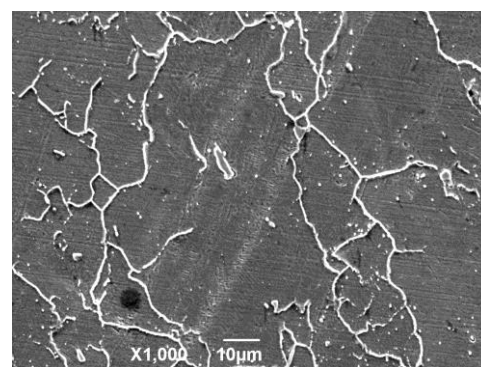
The pipes of Super304H austenitic stainless steel that in the sizes of  $\Phi 44.5\text{mm} \times 9\text{mm}$  were used as base metals. Friction welding was carried out using a continuous drive friction welding machine. During the friction welding operations, the friction welding parameters were set to the following combinations: a friction speed of 1500 rpm, a friction pressure of 150 MPa, an upset pressure of 200 MPa and a friction time of 5s. These specimens were heated respectively in a tube furnace at 625 °C for 0h, 500h, 1000h, 1500h, 2000h, 2500h, and 3000 h, respectively. The microstructures of the aged samples were observed and analyzed by using a scanning electron microscopy (SEM) equipped with an energy dispersive spectroscopy (EDS) system. Vickers hardness values of the joints were tested using a Vickers hardness testing instrument with a loading of 5 kg and an enduring time of 15 s.

## 3. Results and Discussion

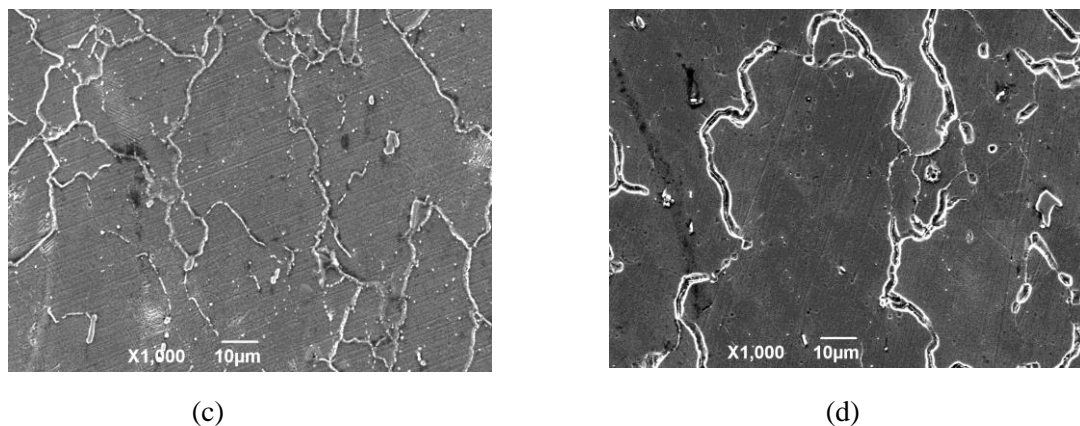
Fig 1- Fig 4 shows precipitates morphology of the welding joints aged for 0h, 1000h, 2000h and 3000 h, respectively. Compared with the conventional fusion welding joints, the friction welded austenitic stainless steel joints were mainly composed of fine equiaxed austenite crystals, and the welded zone has a narrow width. The friction welding was solid state welding techniques, the melting process did not occur in the welded zone and heat affected zone. So the heat input during the friction welding process was much less than the heat input during the fusion welding process. So the friction welded joints with the small grains showed excellent microstructure [7]. It is known that Super304H is famous for its excellent thermal stability. After aged at 625 °C, the austenitic grain size change of the welding joints was not detectable. When the sample was not aged, the precipitated phases that composed of fine spherical particles were formed occasionally, and were mainly distributed along the grains boundary. There were very few precipitated phases in the interior of grains. When the sample was aged for 1000h, it can be observed more precipitated phases distributed along the grain boundary, and the shape of precipitated phases include the lath-like and fine spherical. The precipitated phases was still less in the interior of grains. When the sample was aged for 2000h, the amount of the precipitated phases increased, the size change of the precipitated phases was not detectable obviously. The precipitated phases began distributed along the grains boundary discontinuously, the number of the precipitated phases in the interior of grains increased. When the sample was aged for 3000h, the precipitated phases distributed along the grains boundary continuously, the number of the precipitated phases in the interior of grains increased obviously and distributed in chains. The aggregation and growth of the precipitated phases were main styles of the microstructure evolution. Some precipitated phases that distributed isolated were coarse and lath-like particles [8].



(a)

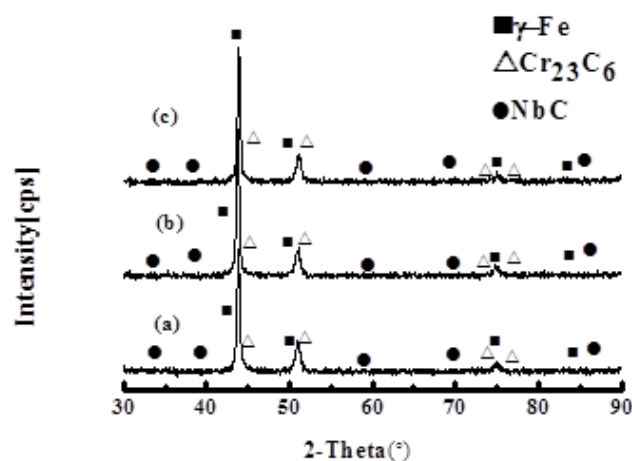


(b)



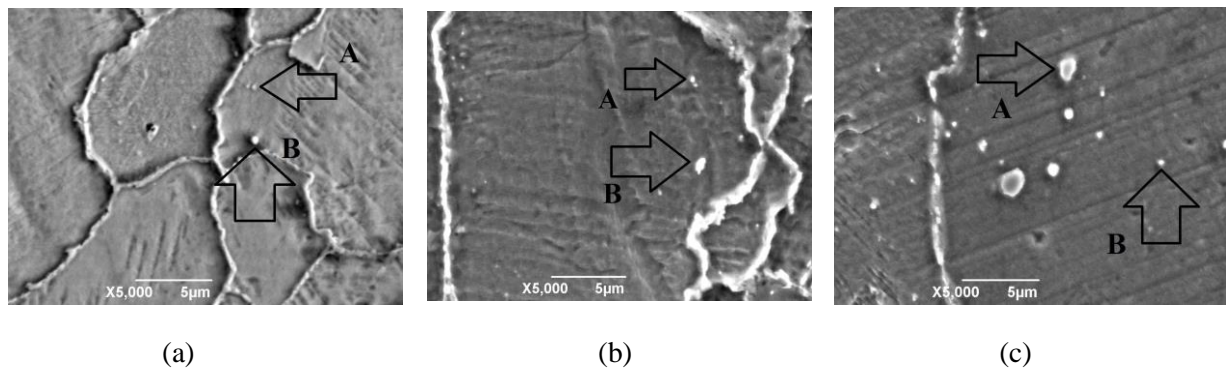
**Figure.1** The precipitated phases of the welding joints: (a) 0h, (b) 1000h, (c) 2000h, (d) 3000h

Fig 2 shows the X-ray diffraction profiles of the welding joints aged for 1000h, 2000h and 3000 h, respectively. It can be sure that the peaks with the highest intensity correspond to the  $\gamma$ -Fe phase and the precipitated phases for all the samples. Therefore, the welded joints after the aging treatment, the main phase is the  $\gamma$ -Fe phase and the precipitated phases are  $\text{Cr}_{23}\text{C}_6$  and NbC. With the increasing of aging time, the lattice parameter and the amount of the precipitated phases  $\text{Cr}_{23}\text{C}_6$  and NbC is increasing. The Cr element has a tendency to form carbides, some C element and Cr element precipitated from matrix and formed the precipitated phases  $\text{Cr}_{23}\text{C}_6$  at the grain boundary preferentially. The X-ray result indicated that the precipitated phases included the Cr and Fe elements, and the Cr element is the major metal element. Therefore, the  $\text{Cr}_{23}\text{C}_6$  is denoted  $\text{M}_{23}\text{C}_6$  precipitated phases. Some N element replaces the element C, and a part of the NbC is converted into Nb(C, N). It is difficult to index the Nb(C, N) due to their lower intensities peaks. Therefore,  $\text{Cr}_{23}\text{C}_6$  and NbC is denoted precipitated phases in the X-ray diffraction profiles. The Super304H welded joints show the excellent creep rupture strength, and the austenite matrix still keep the fine microstructure. This could be attributed to the dispersively distributed of the strengthening phase Nb(C, N) and NbC in the interior of grains, which can effectively pin dislocations and grains boundaries, than impede the movement of dislocations and grains boundaries. The Nb(C, N) and NbC became the core of the subsequent precipitation of  $\text{Cr}_{23}\text{C}_6$ , which avoids the precipitation of  $\text{Cr}_{23}\text{C}_6$  and the depletion of Cr at the grain boundaries. So the precipitation of Nb(C, N) and NbC improves the resistance to intergranular corrosion of the welded joints.



**Figure.2** XRD patterns of the welding joint after aging for different time(a)1000h; (b)2000h; (c)3000h;

Fig 3 shows the precipitated phase of the welding joints aged for different times with EDS to show the elements of the precipitated phases. Table 1 shows the EDS analysis results of the precipitated phases at positions shown in Figure 3. Fig 3 (a) shows the shape of precipitated phases of welded joints aged for 1000h, the precipitated phase A is Nb(C, N) and the precipitated phase B is  $\text{Cr}_{23}\text{C}_6$ . It can be observed some fine  $\text{Cr}_{23}\text{C}_6$  and Nb(C, N) distributed along the grains boundaries and in the interior of grains. With the increasing of the aging temperature, the size and amount of  $\text{Cr}_{23}\text{C}_6$  increase continuously. Fig 3 (b) shows the shape of precipitated phases of welded joints aged for 2000h, the precipitated phase A is Nb(C, N) and the precipitated phase B is  $\text{Cr}_{23}\text{C}_6$ . The amount of  $\text{Cr}_{23}\text{C}_6$  and Nb(C, N) along the grains boundaries and in the interior of grains increased, the size change of Nb(C, N) was not detectable obviously. Fig.3(c) shows the shape of precipitated phases of welded joints aged for 3000h, the precipitated phase A is  $\text{Cr}_{23}\text{C}_6$  and the precipitated phase B is Nb(C, N). The  $\text{Cr}_{23}\text{C}_6$  distributed along the grains boundaries continuously, and the Nb(C, N) distributed in the interior of grains in chains. The aggregation and growth of  $\text{Cr}_{23}\text{C}_6$  were obviously. Some  $\text{Cr}_{23}\text{C}_6$  that distributed isolated were coarse and lath-like.



**Figure.3** The shape of precipitated phases of welded joints: (a)1000h; (b)2000h; (c)3000h;

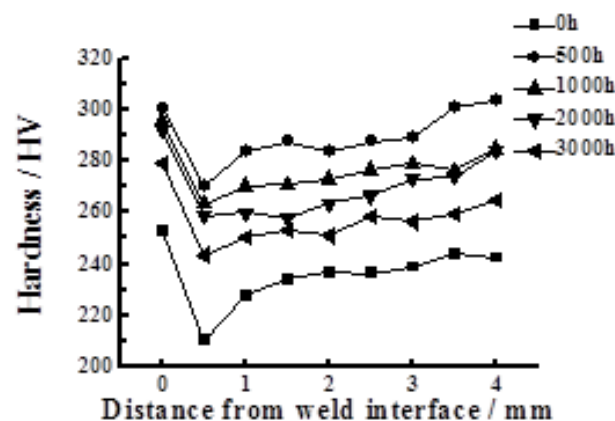
**Table 1** The energy spectrum analysis of the precipitated phase

	C	Cr	Fe	Ni	Cu	Nb
The precipitated phase A (aged for 1000h)	10.08/45.84	1.23/1.29	1.47/1.44	0.34/0.32	0.06/0.06	86.81/51.05
The precipitated phase B (aged for 1000h)	13.55/44.55	16.26/12.35	42.97/30.37	4.19/2.82	0.63/0.39	22.40/9.52
The precipitated phase A (aged for 2000h)	14.82/49.3	12.05/9.56	26.48/19.56	2.44/1.72	1.19/0.76	43.01/19.10
The precipitated phase B (aged for 2000h)	15.13/45.26	18.19/13.00	51.65/34.35	5.66/3.58	0.40/0.23	8.97/3.59
The precipitated phase A (aged for 3000h)	25.04/65.83	11.62/7.05	22.65/12.81	2.06/1.11	0.49/0.24	38.14/12.96
The precipitated phase B (aged for 3000h)	11.83/49.94	1.72/1.68	2.21/2.01	0.29/0.25	0.73/0.69	83.21/45.42

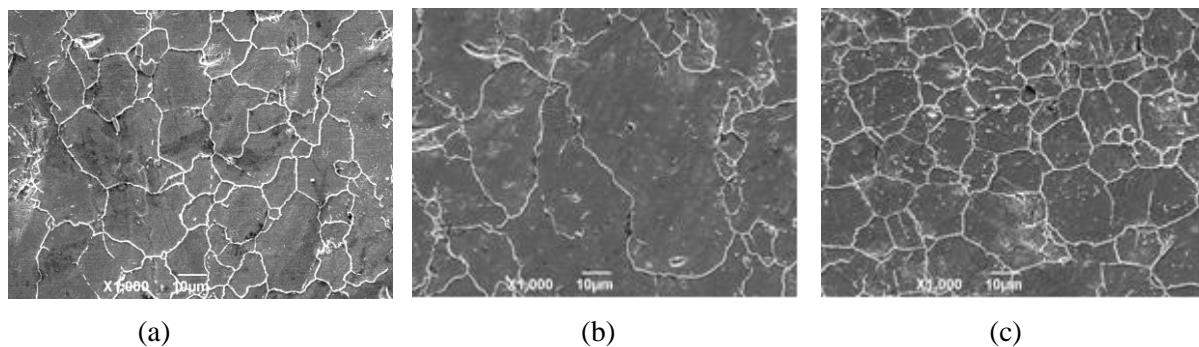
Fig 4 shows the Vickers hardness variations in the direction perpendicular to the weld interface. Fig 5 shows the austenite grains size of the welding zone, the heat affected zone and the base metal. Because the size of austenite grains has a great influence on the Vickers hardness, the Vickers hardness decrease with the increasing of the austenite grains size. Compared with the conventional fusion welding joints, the welded zone of the friction welded joints with a narrow width were mainly composed of fine equiaxed austenite crystals. Furthermore, the strain hardening effect during the friction process at the welded zone is another reason. So the maximum Vickers hardness value is obtained at the welded zone. The friction welding was solid state welding techniques, the melting process did not occur in the welded zone and the heat affected zone. But, the grains size of the heat



affected zone was the larger than the remaining parts of the welded joints, because that the heat input during the friction welding process. So, the minimum Vickers hardness value is obtained in the heat affected zone. Fig 4 depicts the Vickers hardness as a function of the aging time. As the aging time increased, the Vickers hardness of the joints increases sharply (<500 h), and then decreases (500-3000 h). In the aging process, the Vickers hardness change of the welding joints is certainly corresponding to the microstructure evolution. Precipitation, aggregation and growth of the precipitated phases are main reasons of the microstructure evolution of the welding joints. In the early aging stage, the strain hardening effect of the  $\text{Cr}_{23}\text{C}_6$  and  $\text{Nb}(\text{C},\text{N})$  may be overwhelming, the Vickers hardness of the joints increased. As the aging time increased continuously, aggregation and growth of the  $\text{Cr}_{23}\text{C}_6$  takes place, the hardening effect of the precipitated phases decreases simultaneously, resulting in the apparent drop of the Vickers hardness.



**Figure.4** The Vickers hardness of the welding joints after aging for different time



**Figure.5** The austenite grains size of the welding joints: (a) the welding zone, (b) the heat affected zone, (c) the base metal

#### 4. Conclusions

In the aging process, the precipitated phase evolution of the friction welded joints was exhibited.  $\text{Cr}_{23}\text{C}_6$  and  $\text{NbC}$  is denoted precipitated phases in the X-ray diffraction profiles. Precipitation, growth and aggregation of the  $\text{Cr}_{23}\text{C}_6$  were the main precipitated phase evolution mechanism. The welded joints shows the excellent creep rupture strength, it could be attributed to the dispersively distributed of the strengthening phase  $\text{Nb}(\text{C}, \text{N})$  and  $\text{NbC}$  in the interior of grains, which can effectively pin dislocations and grains boundaries. The maximum Vickers hardness value is obtained in the welding zone. As the aging time increased, the Vickers hardness of the joints increased in the initial stage, than decreased after aged for 500 h.

#### Reference

- [1] Zielinski A. Structure and properties of Super 304H steel for pressure elements of boilers with ultra-supercritical parameters[J]. *Journal of Achievements in Materials and Manufacturing Engineering*, 2012, 55(2): 403-409.
- [2] Xinmei Li, Yong Zou, Zhongwen Zhang, et al. Microstructure Evolution of a Novel Super304H Steel Aged at High Temperatures[J]. *Mater Trans*, 2014, 608: 164-173.
- [3] Indrani S, Amankwaha E, Kumara S, et al. Microstructure and mechanical properties of annealed SUS 304H austenitic stainless steel with copper[J]. *Mater Sci Eng A*, 2011, 528: 4491-4499.
- [4] Li P, Li J, Li X, et al. A study of the mechanisms involved in initial friction process of continuous drive friction welding[J]. *Journal of Adhesion Science and Technology*, 2015, 29(12): 1246-1257.
- [5] Li X, Li J, Liao Z, et al. Effect of rotation speed on friction behavior and radially non-uniform local mechanical properties of AA6061-T6 rotary friction welded joint[J]. *Journal of Adhesion Science and Technology*, 2018, 32(18): 1987-2006.
- [6] Li X, Li J, Liao Z, et al. Microstructure evolution and mechanical properties of rotary friction welded TC4/SUS321 joints at various rotation speeds[J]. *Materials & Design*, 2016, 99: 26-36.
- [7] Xinmei Li, Yong Zou, Zhongwen Zhang, et al. Microstructure Evolution of a Novel Super304H Steel Aged at High Temperatures[J]. *Mater Trans*, 2014, 608: 164-173.
- [8] Guohong C, Qi Z, Junjian L, et al. Microstructures and mechanical properties of T92/Super304H dissimilar steel weld joints after high-temperature ageing[J]. *Mater design*, 2013, 44: 469-475.