CHAPTER 3

Experimental Procedure

3.1 Substrate Material

The substrate used in this study was solution treated DSS JIS SUS329J1 supplied by Ehime University in Japan. The chemical composition of DSS was confirmed using Shimadzu OES-5500II Optical Emission Spectrometer and shown in table below.

Table 3.1 Chemical composition of DSS

Element	C	Si	Mn	P	S	Ni	Cr	Mo	Fe
Wt %	0.06	0.42	0.30	0.03	0.06	4.18	24.5	0.49	Bal

The as-received DSS was thermo-mechanically treated by solution treating at 1573 K for 1 hour followed by water quenching. It was then cold rolled at 75% of reduction area to a plate with 8 mm thickness. The schematic diagram of the thermo-mechanical treatment of DSS is shown in Figure 3.1. Ekabor powder was used as boronizing agent. In general, the commercial boronizing mixture contains B_4C as donor, KBF_4 as an activator and SiC as diluents, which control the boronizing potential of the medium.

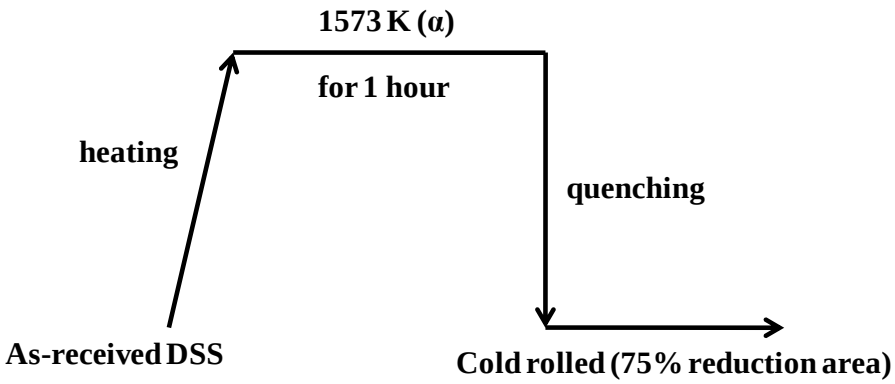

Figure 3.1 Schematic diagram of thermo-mechanical treatment process of DSS

3.2 Material Preparation

The as-received specimen was cut by using a wire electron discharge machine (EDM) (type Sodick), into the size of 15 × 10 × 8 mm as shown in Figure 3.2.

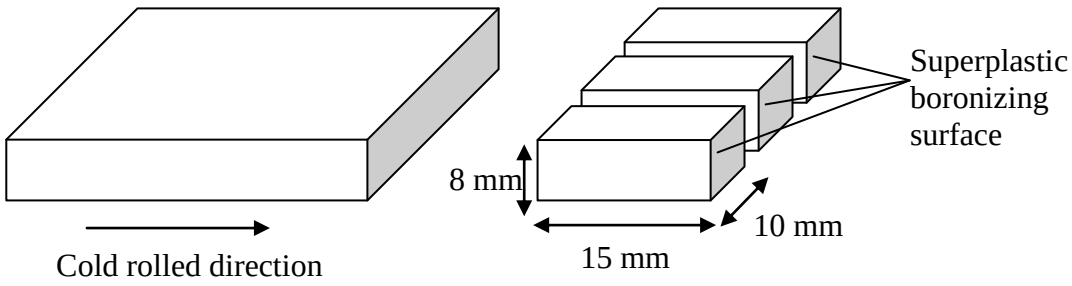

Figure 3.2 Sample preparation

To prepare for SPB process, the surfaces were ground with emery paper of grit 100 and 240 to remove the oxide layer formed during cutting. Then, the surfaces were pickled with alcohol to remove grease and contaminants.

To evaluate the surface microstructure, the surface was ground by using grinding machine with emery paper grade 100, 240, 800 and 1500. The grid paper started with coarsest, 100 to the finest, 1500. The surface to be boronized was held downwards until a flat and smooth surface with parallel line was produced. Then polished the surface to remove scratches and deformation from grinding until a flat and mirror-like surface was obtained. A special etchant following the proportion as mentioned by Voort (1984) in his Metallography book was used as etching reagent to observe the microstructure under optical microscope. The etching reagent is hydrochloric acid (HCl) saturated with ferric chloride ($FeCl_3$) activated with small amount of nitric acid (HNO_3). The etching reagent was swab to the surface of the sample about 30 to 60 seconds. It revealed grain structure of DSS.

3.3 Design Boronizing Crucible

Stainless steel 330 supplied by Ehime University in Japan was selected as material to fabricate the device and crucible used for boronizing. The fabrication process using the power saw and lathe machine. Stainless steel 330 is well known of its corrosion resistance, high ductility, strength and hardness. The melting point of this steel is 1673 K to 1723 K. All of these properties are very suitable for boronizing process. The design of crucible dimension is shown in figure below.

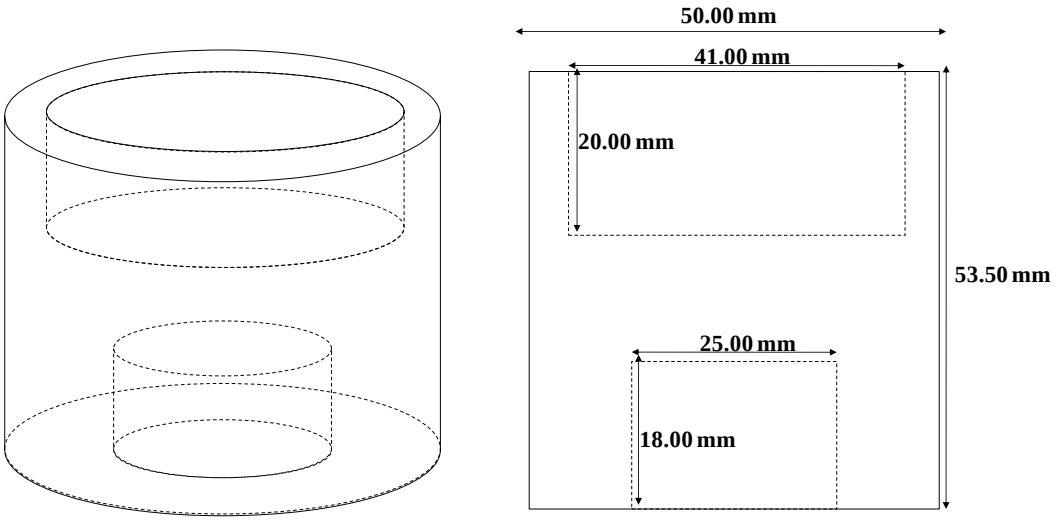

Figure 3.3 Schematic diagram of boronizing crucible

3.4 Superplastic Boronizing Process

SPB was conducted using compression testing machine (Instron) that is equipped with specially designed die and high-temperature furnace under controlled gas atmosphere. Argon gas was used as the controlled gas throughout the process to offer a controlled atmospheric which would avoid oxidation. Figure 3.4 shows the schematic diagram of the experimental apparatus.

The crucible was filled with 10 g boronizing powder then the crucible was tapped to get a dense powder pack of 5 mm thickness. The amount of boronizing powder used to fill in the crucible was weighed using the electronic weighing instrument. The specimen was placed in the middle of the crucible and lay on top of the boron powder. Placed the crucible on top of lower die then inserted the crucible and lower die into the furnace. After that, adjusted the moveable crosshead until the upper die touched the specimen, heated the crucible with specimen and boron powder to

preset temperature, 950°C, then hold the temperature for one hour, compressed the specimen subsequently at 2×10^{-4} s^{-1} strain rate and strain value of 0.2, 0.4 and 0.6. Water was allowed to flow towards the pulling rod to prevent the system from overheating. Lastly, cooled the specimen in controlled atmospheric environment. Repeated this experiment at 1000°C and 1050°C with similar amount of boron powder. Figure 3.5 shows schematically the SPB process step. Table 3.2 tabulates the experimental conditions for SPB study.

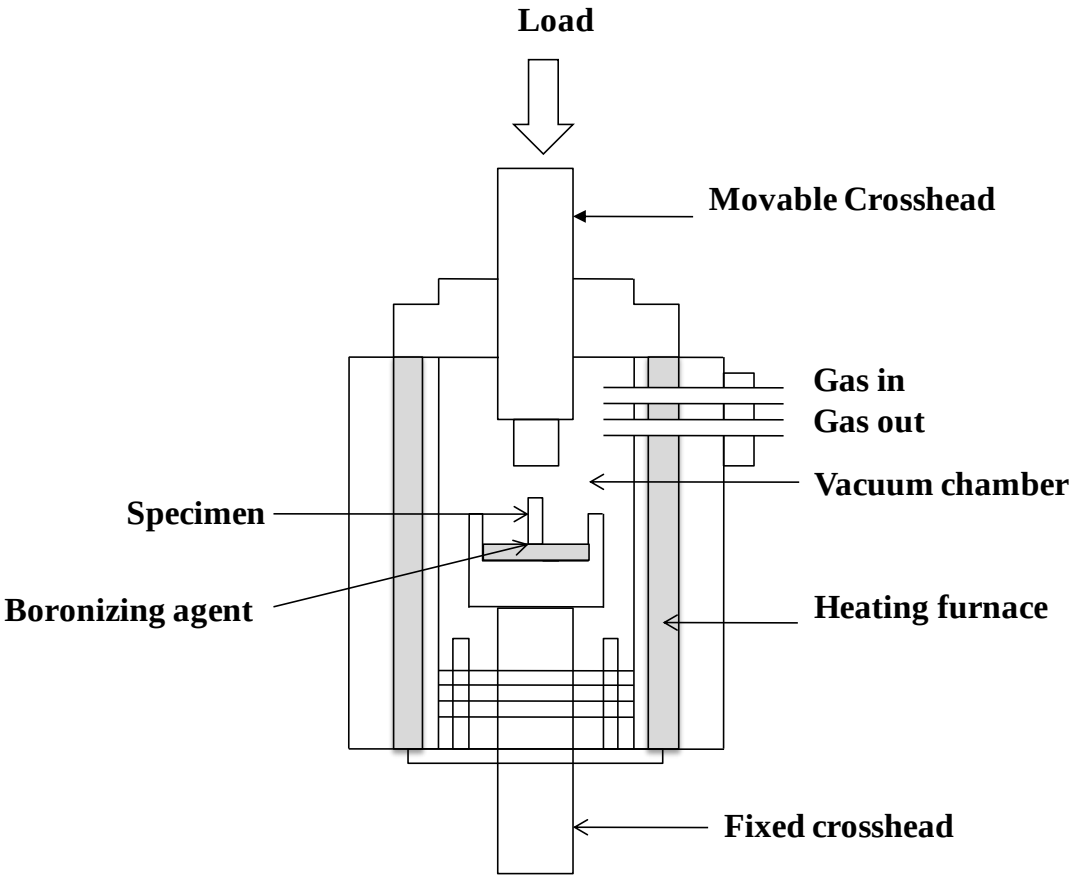

Figure 3.4 Schematic diagram of experimental apparatus

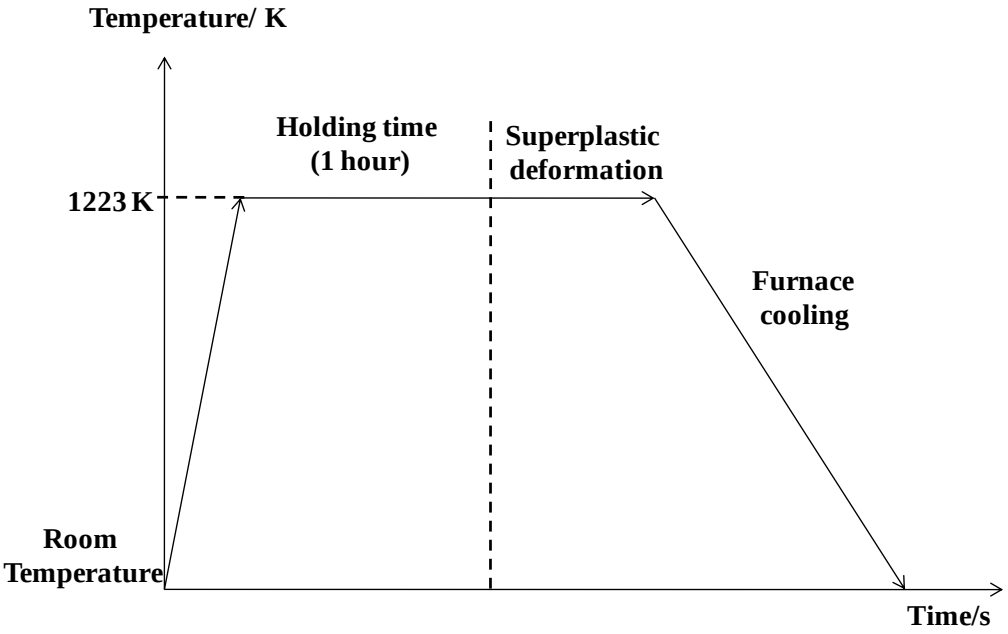

Figure 3.5 Illustration of boronizing experiment

Table 3.2 Experimental conditions for SPB study

Temperature (K)	Strain rate (s^{-1})	Time (s)	Strain
1223	2×10^{-4}	1000	0.2
		2000	0.4
		3000	0.6
1273	2×10^{-4}	1000	0.2
		2000	0.4
		3000	0.6
1323	2×10^{-4}	1000	0.2
		2000	0.4
		3000	0.6

3.5 Characterization Methods

Several characterization techniques were utilized to study the macro and microstructure of the boronized specimen. X-ray diffraction analysis was conducted to confirm the presence of boride phase on the boronized layer. The microstructure of the work piece was studied using the optical microscope and scanning electron microscope (SEM). The surface hardness was also measured before and after boronizing process.

3.5.1 Optical Microscope

The use of visible light and optical lenses to enlarge the image of the small object provides a wealth of information. Optical microscopy analysis enables materials surface treatments to be identified and characterized. In a reflected light microscope, the specimen is illuminated by frontal lighting, which is accomplished by means of a small plain-glass reflector placed inside the tube of the microscope. The optical microscope is used in microstructural analysis and determination of boronized layer thickness and morphology. The optical microscope Zeiss Axiotech with maximum 1000 times enlargement was connected by a Panasonic digital camera model WV-CP410 to an image analyzer MSQ software version 6.5. The digital camera captures and displays the image from the optical microscope directly on the computer screen.

Figure 3.6 Optical microscope with image analyzer system

3.5.2 Scanning Electron Microscopy

Scanning electron microscopes (SEM) function exactly as their optical counterparts except that they use a focused beam of high-energy electrons instead of light to image the specimen and gain information as to its structure and composition. In this study, a Philips model XL40 SEM was used at an operating voltage of 20 kV to examine the microstructure of the specimens before and after boronizing. There are many advantages to using the SEM instead of a light microscope.

While SEM imaging a specimen, the electrons interact with the atoms and make the specimen producing signals of secondary electrons, back-scattered electrons, X-rays, light (cathodoluminescence), etc., that contain information about the specimen surface's properties. Specimen preparation is necessary before taking SEM images because SEM operates in a vacuum and rely on electric fields to work. Preparation of the specimen is

relatively easy since most SEMs only require the specimen to be conductive. The specimen to be analyzed is polished and etched. Mount the specimen on the specimen support stub with conductive carbon cement. The prepared stub is then placed onto the SEM stage in the microscope chamber for analysis. Once the specimen is inserted into the microscope, operator will generate the beam, make the appropriate adjustments on accelerating voltage, magnification, contrast and brightness, and then begin viewing.

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time. The SEM also produces images of high resolution, which means that examination of closely spaced features at a high magnification is possible. The combination of higher magnification, larger depth of focus, greater resolution and ease of specimen observation make the SEM one of the most heavily used instruments in research areas today. Figure 3.7 shows the scanning electron microscope (Ismail, 2004).

Figure 3.7 Scanning electron microscope

3.5.3 Microhardness Test

The microhardness test method according to ASTM E-384 specifies a range of loads using a diamond indenter to make an indentation, which is measured and converted to a hardness value. It is very useful for testing on a wide type of materials as long as test samples are carefully prepared.

After boronizing process, the hardness of treated surface and cross section of boronized samples from the boronized area to the core of the specimen was measured using Micro hardness Tester model MVK H2 with load of 200 g (2N). Two diagonals of the indentation left in the surface of the boronized sample after removal of the load. The diagonals were measured then the hardness reading was calculated and obtained automatically.

Figure 3.8 Microhardness tester

3.5.4 X-ray Diffraction

X-ray diffraction techniques find the geometry or shape of a molecule based on the elastic scattering of X-rays from structures that have long-range order. X-ray scattering techniques are family of non-destructive analytical techniques that reveal information about the crystallographic structure, chemical composition, and physical properties of materials and thin films. These techniques observe the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy (Internet source 6).

X-ray diffraction (XRD) spectra of the specimens before and after boronizing were determined using a Philips X'Pert MPD PW3040 XRD with CuKα radiation at 1.54056 Å X-ray wavelength. The specimen were scanned from 10° to 80° 2θ angle at a step size of 0.020 and a count time of 1.5 s at each step (Ismail, 2004). Figure 3.9 shows the XRD machine.

Figure 3.9 XRD machine