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Microstructural characterization and mechanical properties of mini specimen of research reactor fuel cladding

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Mini specimens of 5052 alloy have been used to study the mechanical behaviour for cladding materials of research reactor. The problem that arises in the manufacture of the mini specimens is the deformation effect after shear cutting process. The objective of this research is to obtain characteristics of mini specimens of 5052 alloy, *i.e.* microstructure, hardness and tensile strength. The results have showen that microstructureof fresh 5052 alloy is not homogeneous, but that of as-rolled 5052 alloy is non-homogeneous for elongated flat along the roll direction. In the 250 °C heat treatment, the grain structure of as-rolled 5052 alloys is increasingly homogeneous with slight grain size changes as the heating time increases. The hardness of 5052 alloy as-rolled after heat treatment ranges from 39.67 - 40.55 VHN. The hardness of as-rolled 5052 alloy is higher than that of fresh 5052 alloy without and post heat treatment. Tensile strength of as-rolled 5052 alloy after heat treatment is relatively the same, *i.e.* 159.50 – 160.27 MPa with a strain of 31.67 – 34.58%. Tensile strength of 5052 alloy after mechanical treatment has higher tensile strength than fresh and as-rolled 5052 alloys. The mechanical treatment of as-rolled 5052 alloy could eliminates the shear cutting effect on the surface of mini specimens.

Keywords: AA 5051, Mini specimen, Shear cutting, Microstructure, Tensile strength

1 Introduction

AA 5052 aluminum alloy is selected as a research reactor fuel cladding material[1] because it has low thermal neutron absorption cross-section, its oxidation resistance and mechanical strength are quite good at temperatures of about 100 °C, it also has high thermal conductivity, and high corrosion resistance at the operating temperature of research reactors. It is easily fabricated, the best welding compared to other aluminum alloys, cheap and easily available in the market[2]. AA 5052 alloy has the microscopic thermal neutron absorption cross-section of 0.23 barn, density of 2.68 g/cm^3, thermal conductivity of 137 W/m.K and melting point of 650 °C. At room temperature, the AA 5052 alloy have tensile strength of 170 MPa, yield strength of 58 MPa, strain of 18 - 21%, and modulus elasticity of 71 GPa[3].

In the research reactor, the fuel cladding is continuously exposed to internal stresses due to fuel swelling, fission products formed and heat of fission reactions. In this regard, the cladding material must have greater strength and ductility than the requirements for the cladding material of the research reactor fuel in order to be able to withstand internal stresses[4]. Therefore, some requirements that must be met to determine the thickness of cladding of a fuel plate include: maximum cladding temperature of 145 °C, maximum meat temperature of 207 °C, the ratio of coolant velocity to its critical velocity between 0.63 to 0.85 and the value of flow instability is greater than 2.67. Based on the requirement, fuel element plate of U_3Si_2/Al with uranium density of 2.96 g/cm^3 using AA 5052 alloy with thickness 0.38 mm has a dimension: 70 cm (length), 7 cm (width), 1.34 mm (thickness)[5].

The quality of nuclear fuel elements can be known from the pre and post irradiation test data which includes non-destructive (NDT) and destructive (DT) test. One of the destructive tests is the tensile test. In the tensile testing of irradiated fuel plate, it is necessary to consider several things: (1) ease of handling because it require a manipulator to operate a tensile test machine in a hotcell, and (2) minimization of waste because it potentially increases radiation exposure and contamination inside the hotcell. One technique used to overcome these problems is the use of mini specimens to determine the mechanical behavior of fuel plate cladding materials. The problem that arises in the manufacture of mini specimens is the deformation on the cut surface of

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material with a punching machine. The shear cutting effect can be removed by heat treatment and cut surface grinding of mini-specimen with SiC sandpaper[6].

Prochaska *et al.* in their study using a mini specimen found that its tensile strength was proportional to the tensile strength of standard specimens, while the strain was more sensitive than the standardized specimen strain as it related to surface roughness and microstructure of mini specimens[7]. Bo Wang *et al.* had conducted a study of AA 5052 alloy as cast which was homogenized at 470 °C for 15 hours with air sample cooling. The sample is then cooled with a reduction of thickness of 15% to 87%. Bo Wang found that the increased reduction of thickness applied to AA 5052 alloy caused the grains severily elongated along the rolling direction and the grains accumulated to increase the strain hardening effect which result in the enhanced strength and decraded plasticity of AA 5052 alloy[8]. Factors that influence the tensile properties of mini specimens are surface roughness, specimen thickness, and radiation damage[9].

Based on the description, it is known that the previous researchers only conducted research related to the effect of surface roughness, thickness and reduction of thickness to the tensile properties of mini specimens of AA 5052 alloys. Accordingly, this research has heat treatment of mini specimens of AA 5052 alloys based on the maximum permissible fuel temperature and mechanical treatment *i.e.* grinding on the cut surface of the specimen to eliminate the shear cutting effect due to the processing of mini specimen on the material fuel cladding behavior. The objective of this research is to obtain the characteristics of AA 5052 mini specimens after heat and mechanical treatment that include microstructure, hardness, and tensile strength. The heat treatment of AA 5052 mini specimens was carried out at a temperature of 250 °C with holding time of 1, and 5 hours. Meanwhile, the mechanical treatment by grinding on cut surface of mini specimens of AA 5052 alloy using 1200 grit sandpaper. The proposed research hypothesis are that surface heating can uniform grain size in microstructures and be expected to increase the ductility and decrease slightly the hardness and the strength of AA 5052 alloy.

2 Experimental Method

The material that used are mini specimens of fresh AA 5052 alloy with dimensions : 12.0 mm (length), 3.01 mm (width), 2.6 mm (thickness) and as-rolled with dimensions : 12.0 mm (length), 3.01 mm (width), 1.4 mm (thickness) product of punching machine[10]. Other materials used are acryfic resin and hardener, sandpaper, polishing cloth, diamond paste, and etching solution for metallographic preparation. Meanwhile, the equipment used in the research is a heating furnace, cutting machine, grinding machine and polish, optical microscope, microhardness Vickers tester, and tensile testing machine.

The stages of research was heat treatment process, metallographic preparation, microstructural observation, micro hardness testing and tensile testing of mini specimens of AA 5052 after heat and mechanical treatment. Samples are heat reated at 250 ^{o}C for 1, 3 and 5 hours. After that the samples are cooled slowly in furnace. The microstructure observations of mini specimens refer to ASTM E3-11, micro hardness testing of mini specimens refer to ASTM E384-16, and tensile testing of mini specimens refer to ASTM B557-15 standard.

Characterization of the AA 5052 mini specimens without heating and after-heating and mechanical treatment was carried out with the following stages: (1) the metallographic specimen is mounted with acryfic resin and hardener, then grinding with 320 to 2000 grit of sandpaper to obtain a flat, scratch-free sample, then polished with 1 μm rough diamond paste to obtain a flat and shiny surface, and subsequent etching with the swab method; (2) observation of specimens microstructure using optical microscope; (3) micro hardness testing of specimens using microhardnes Vickers tester with 200 gf load. Meanwhile, the tensile test on the mini specimen was performed by the tensile test machine with a load of 5 kN to obtain tensile strength, yield strength and strain of AA 5052 alloy. Data obtained from the characterization of mini specimens were analyzed to determine the characteristics of AA 5052 alloy after heat and mechanical treatment *i.e.* microstructure, hardness and tensile properties of AA 5052 alloy and tensile properties of AA 5052 alloy after mechanical treatment.

3 Results and Discussion

3.1 Microstructure

In this research, the heat treatment process on mini specimens of AA 5052 alloys was carried out at a temperature of 250 ºC with 3 (three) retention times variations of 1, 3 and 5 hours, and cooling the

specimens in the heating furnace. Figure 1 shows the microstructure of fresh AA 5052 alloy specimens with thickness of 2.6 mm before and after heat treatment. Figure 1a shows the grains structure of AA 5052 alloy in the form mixed grains *i.e.* coarser equiaxial, elongated flat and smaller equiaxial grains. Because the microstructure of fresh AA 5052 alloy without heat treatment has 3 (three) different grain forms then high residual stress in the alloys. This affects the behavior of the alloy that is quite hard, as reflected in the hardness value of 44.94 ± 4.19 VHN. Figure 1b shows a microstructure of coarser equiaxial, elongated flat and smaller equiaxial grains with larger sizes relatively compared to the size of the equiaxial grains of Fig. 1a, while the microstructures in Fig. 1c are mixed grains *i.e.* coarser equiaxial and elongated flat, and in Fig. 1d occurs grain growth of equiaxial and elongated flat grains.

Microstructures of AA 5052 alloy after heat treatment has the same relative grain shape so that with the increase of heating time will occur grain incorporation into equiaxial and elongated flat grain coarser compared to the structure of AA 5052 alloy grains without heating. AA 5052 alloy after heat treatment is relatively soft because of its relatively small hardness decline. This is due to the enormous role of softening by the recovery mechanism, particularly the dynamic recovery so that the driving force is low and insufficient to induce dynamic recrystallization[11].

Figure 2 shows the microstructure of as-rolled AA 5052 alloy specimens with thickness of 1.4 mm after heat treatment. The as-rolled AA 5052 alloys have typical flat elongated grains structure with α (Al) matrix and a large amount of second phases distributed along the rolling direction (Fig. 2a). The

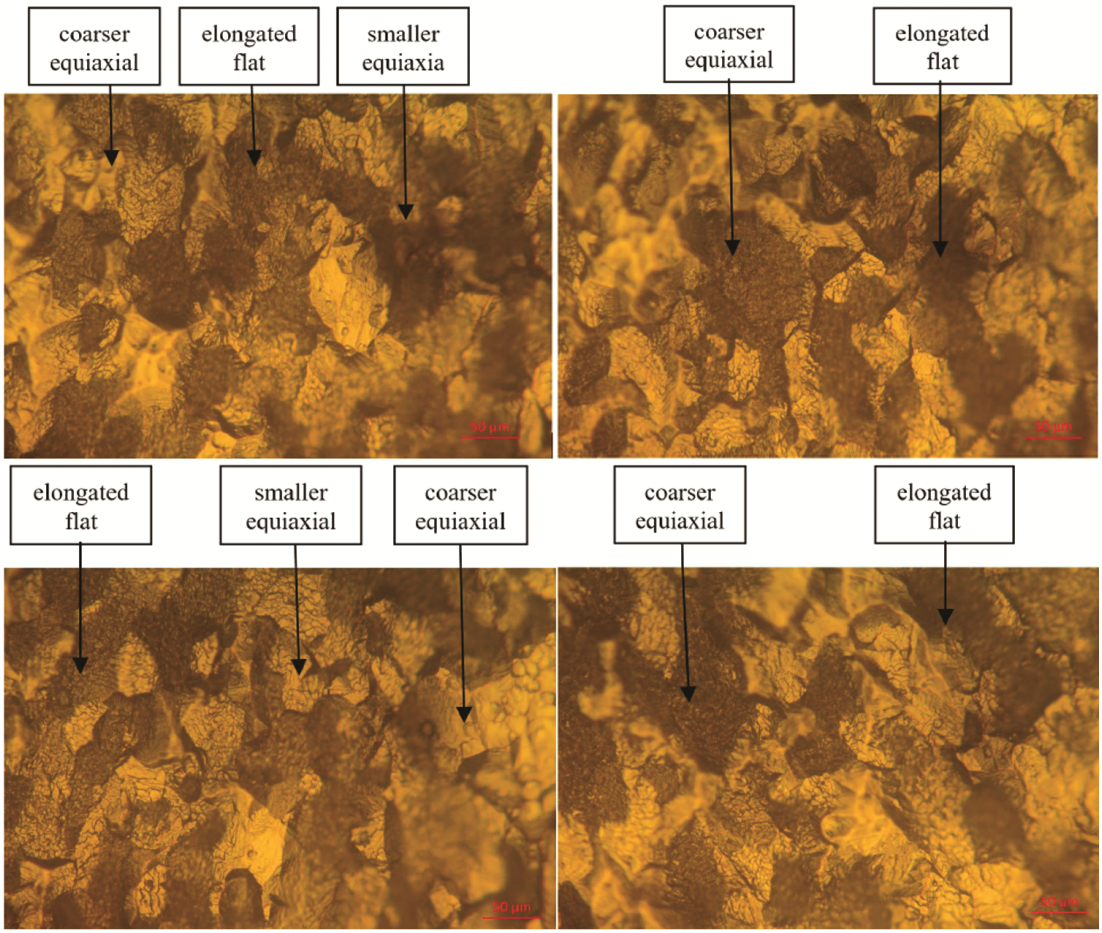

Fig. 1a — Optical micrograph of fresh AA 5052 alloy with thickness of 2.6 mm before treatment heating.
Fig. 1b — Optical micrograph of fresh AA 5052 alloy with thickness of 2.6 mm after heating of 250 °C and holding time of 1 hour.
Fig. 1c — Optical micrograph of fresh AA 5052 alloy with thickness of 2.6 mm after heating treatment of 250 °C and holding time 3 h.
Fig. 1d — Optical micrograph of fresh AA 5052 alloy with thickness of 2.6 mm after heat treatment of 250 °C and holding time 5 h.

grains grow obviously after heat treatment. It can also be seen that some large second-phase particles and most of the dendrites are dissolved and disappear in the as-homogenized alloy from Figs 2 (b), (c) and (d).

When a AA 5052 alloy is cold-worked by plastic deformation, a small portion of the mechanical energy expended in deforming the metal is stored in the specimen. This stored energy resides in the crystals as point defects (vacancies and interstitials), dislocations, and stacking faults in various forms and combinations. Therefore, a cold-worked specimen, being in a state of higher energy, is thermodynamically unstable. With thermal activation, such as provided by annealing, the cold-worked specimen tends to transform to states of lower energies through a sequence of processes with microstructural changes. During recovery, accumulated strain is relieved to some extent by microstructural and submicroscopic rearrangements, but the grains are not entirely strain-free[12]. At higher temperatures, strain-free grains are created during the restoration process of recrystallization. Along with the microstructural changes, the properties of the specimen also change correspondingly.

3.2 Hardness

The results of the micro hardness testing on the mini specimen of AA 5052 alloy before and after heat treatment are shown in Table 1. It is seen that the hardness of AA 5052 alloy after heat treatment is lower than before and after the heat treatment of fresh or as-rolled AA 5052 alloy. The AA 5052 alloys as-rolled have a higher hardness value than AA 5052 fresh alloy before and after heat treatment.

Table 1 — Hardness value of AA 5052 alloy before and after treatments.

Specimen thickness, mm	Micro hardness, VHN			
	Heating of 250 °C with holding time			
	Without heating	1 hour	3 hours	5 hours
2.62 ± 0.02	44.94 ± 4.19	37.77 ± 1.46	39.42 ± 4.88	35.26 ± 0.27
1.42 ± 0.01	52.85 ± 2.78	40.55 ± 3.15	39.68 ± 1.59	39.67 ± 1.73

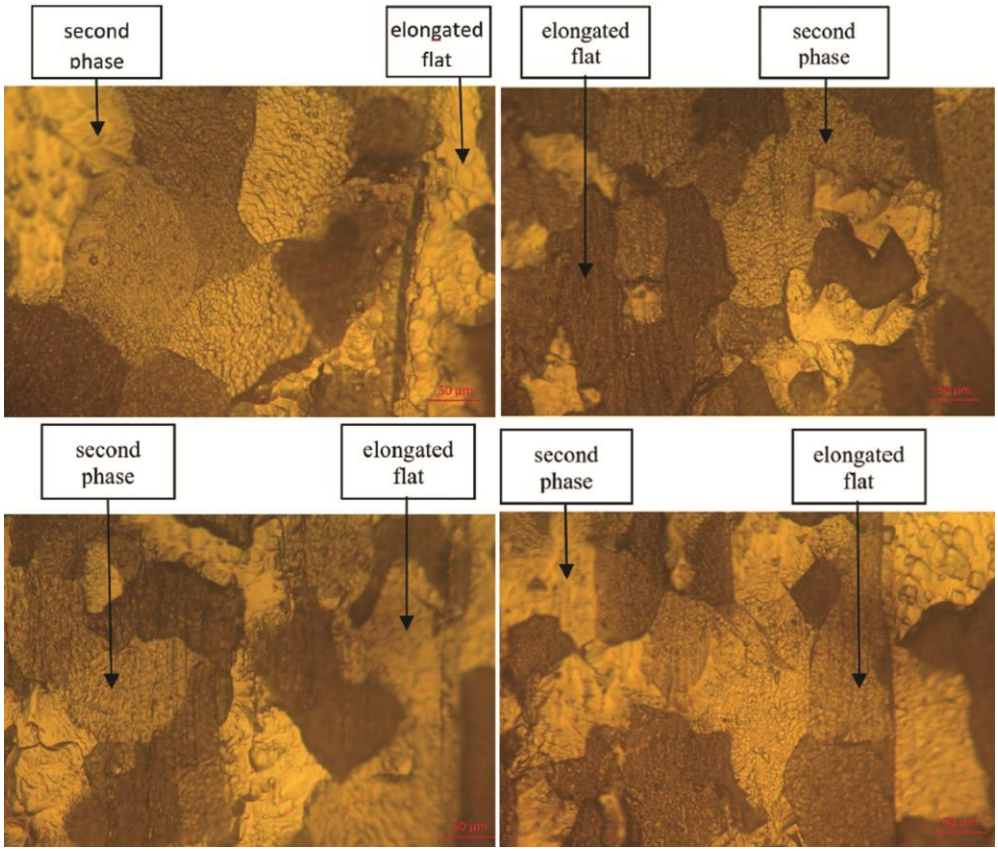

Fig. 2a — Optical micrograph of as-rolled AA 5052 alloy with thickness of 1.4 mm without heat treatment.
Fig. 2b — Optical micrograph ofas-rolled AA 5052 alloy with thickness of 1.4 mm after heat treatment of 250 °C and holding time of 1 h.
Fig. 2c — Optical micrograph ofas-rolled AA 5052 alloy with thickness of 1.4 mm after heat treatment of 250 °C, holding time of 3 h.
Fig. 2d — Optical micrograph ofas-rolled AA 5052 alloy with thickness of 1.4 mm after heat treatment of 250 °C, holding time of 5 h.

Table 2 — Mechanical properties of AA 5052 alloy before and after heat and mechanical treatment.

Specimen thickness, mm	Treatment	Tensile strength (UTS), MPa	Yield strength (σ_Y), MPa	Strain (e), %
2,62 ± 0.02	without heating	169.25 ± 0.01	131.20	26.63
(Fresh)	250 °C, 1 hour	154.39 ± 1.18	75.87	38.42
	250 °C, 3 hours	153.08 ± 0.57	92.66	37.13
	mechanical	174.92 ± 3.10	146.56	24.17
1.42 ± 0.01	without heating	167.08 ± 4.79	101.76	21.21
(As-rolled)	250 °C, 1 hour	160.27 ± 0.49	89.30	34.58
	250 °C, 3 hours	159.50 ± 0.25	89.49	31.67
1.31 ± 0.01	mechanical	180.09 ± 3.82	165.83	22.25

AA 5052 alloy as-rolled after heat treatment at 250 °C with a holding time of 1 - 5 hours have the same relative hardness value. This indicates that the heat treatment process against on mibi specimens of the AA 5052 alloy are the recovering process of the grains structure of AA 5052 as-rolled alloy due to metal processing and mini-specimen preparation. The recovery process of AA 5052 alloy is carried out at temperatures below 0.4 melting point of alloy metal, in this case 250 °C. This recovery process aims to uniform the shape and size of the grain so that residual stress in microstructure of AA 5052 alloy to a minimum. If this is achieved then the hardness of AA 5052 alloy is relatively the same[13].

3.3 Mechanical Properties

The tensile test results of mini specimen of AA 5052 alloy after heat and mechanical treatment were shown in Table 2. The AA 5052 alloy with thickness 1.4 mm and 2.6 mm are heat treated, respectively at 250 °C and holding time of 1 and 3 hours.

In the specimens with a thickness of 1.4 mm it appears that the tensile strength of as-rolled AA 5052 alloy is 167.08 MPa, after heating of 250 °C is 160.27 MPa for holding time of 1 hour and 159.50 MPa for holding time of 3 hours. Tensile strength of AA 5052 alloy after heating is lower and its ductility is higher than AA 5052 alloy as-rolled. This is caused to heating can give rise to increase of grain size. According to the Hall−Petch relation $\sigma_y \propto d^{-1/2}$, grain size *d* plays an important role in improving the tensile strength[14]. Meanwhile, the tensile strength of AA 5052 alloy after heating of 250 °C decrease slightly for longer retention time. This is caused occurrence of recovery process in microstructure of AA 5052 alloy so elongated grains have relative the same and homogeneous size. After heating at 250 °C for 1 hour, the elongation of the mini specimen is enhanced from 21.21 % to 34.58 %., and decrease slightly from 34.58 % to 31.67% on heating for 3 hours. This is due to the heating grain growth occurs so that the grains grow larger and the impact there is a decrease in tensile strength. However, at the same heating temperature there was a decrease in the elongation caused by increasing holding time at same temperature, the secondary phase is coarser, which is bad for improving strength.

Tensile strength of the AA 5052 alloy after mechanical treatment (= 180.09 MPa) has higher than tensile strength of fresh and as-rolled AA 5052 alloys. This proves that the mechanical treatment can be eliminate the shear cutting effect on the surface of the AA 5052 alloy mini specimens[15].

4 Conclusions

Microstructures of fresh AA 5052 alloy form coarser equiaxial and elongated flat and fine exuiaxial grains, while of as-rolled AA 5052 alloy are non homogeneous flat elongated grains along the direction of the roll. In the 250 °C heat treatment, the grain structure of as-rolled AA 5052 alloy is increasingly homogeneous with slight grain size changes as the heating time increases.

The hardness of as-rolled AA 5052 alloy after heat treatment is relatively similar, ie 39.67 - 40.55 VHN. The hardness of as-rolled AA 5052 alloys is higher than fresh AA 5052 alloy without and after heat treatment.

Tensile strength of as-rolled AA 5052 alloy after heat treatment is relatively the same ie 159.50 – 160.27 MPa with strain of 31.67 – 34.58%. Under the same heat treatment conditions, the tensile strength of as-rolled AA 5052 alloy is higher and the strain is lower than fresh AA 5052 alloy. Tensile strength of AA 5052 alloy after mechanical treatment has higher

tensile strength than fresh and as-rolled AA 5052 alloys.

The heat treatment of as-rolled AA 5052 alloy mini specimens is a recovery process, while the mechanical treatment to eliminate the shear cutting effect on the surface of the AA 5052 alloy mini specimens.

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