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Influence of Thermal Cycling Temperatur on the Recrystallization of Cold Rolled Stainless Steel 316L

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Abstract. Investment casting of an orthopedic implant plate based on stainless steel 316L was considered an economical process. Nevertheless, the mechanical properties of the investment casting product were found to be inferior as compared to the implant plate fabricated with other methods such as forging due to their differences in the microstructure. Investment casting mostly produced coarser grain as compared to those with forging or rolled process. In order to improve their mechanical properties, cold-rolling followed by a repetitive thermal cycling process is proposed. The goal is to generate finer grain size through recrystallization process leading to nucleation of new grain during the thermal cycling process thus increasing their strength. Stainless steel 316L was cold-rolled to 52% reduction in thickness and this process generate stored strain energy in the form of dislocation density in the material. The thermal cycling treatment performed within several cycles after cold rolling enabling gradual disperse of stored strain energy that facilitates the recrystallization process that initiates new grain formation. The short holding time within several cycles limits the grain growth that normally occurs during annealing. It was found that thermal cycling treatment at a temperature of 950 °C for 35 seconds within four cycles led to the formation of finer grain size of 22 μm on average as compared to the initial investment casting average grain size of 290 μm. The hardness also increases to 253 $HV_{0.3}$ in this condition as compared to 155 $HV_{0.3}$ of investment casting products. Lower thermal cycling temperature than 950 °C during the test did not result in grain refinement thus indicating that strain energy relieves were not enough to aid the recrystallization process.

Introduction

In a case where bone fracture happens, an orthopedic surgeon occasionally installs temporary orthopedic implant plates to hold and support the broken bone after returning the bone into their original position. Austenitic stainless steel 316L is commonly selected due to its high strength, high fracture toughness, high corrosion resistance, and good ductility. It is also more economical options as compared to other biometal implant materials such as titanium alloy or cobalt alloy [1]. The 316L implant can be manufactured by investment casting, CNC machining or forging process. The investment casting 316L is more economical but generally produces lower mechanical properties as compared to other manufacturing methods due to the existence of internal defects such as porosity or impurities.

Several treatments such as cold rolling (CR), strain-induced metastable α' martensite (SIM) generation, grain size refinement through cold rolling-annealing (CRA), severe plastic deformation (SPD) and thermo-mechanical treatment (TMT) can be employed to austenitic stainless steel to improve their mechanical properties [2][3]. These methods mostly utilize the metastable condition of austenitic phase that can be transformed into α' martensite during cold deformation and recrystallization of new finer grain during subsequent annealing or thermomechanical processing. Nevertheless, the transformation to SIM during cold deformation is much more difficult on those highly stable austenitic stainless steel such as 304L and 316L. Thus the treatment can only depend on the availability of stored strain energy generated after cold deformation in the form of dislocation density. The availability of stored strain energy will accelerate recrystallization kinetics during thermal cycling procedures by imposing strain heterogeneity in the microstructures [4]. The higher the stored energy, the easier the recrystallization to take place within lower temperatures due to many stored energy being released during exposure to elevated temperature [5]. Repetitive thermal process after cold deformation will gradually release stored strain energy thus recrystallization of newer grain can occurs in each cycle [5]. Nanda [6] indicates that after severe cold rolling of 70% thickness reduction followed by repetitive thermal cycling at 900° C, the grain size of 316L was reduced to 0.8 - 1.2 µm from 90-120 µm previously. The tensile strength also improved from 590 MPa to 1220 MPa. In this research, a lower cold rolling reduction of 52% followed by repetitive thermal cycling temperature at 850, 900 and 950 °C was investigated in order to found at which thermal cycling temperature the recrystallization of new grain begins to emerge in relation to their stored strain energy.

The amount of stored strain energy in steel after the cold-rolling process can be calculated as ρ dislocation density (m^{-2}) influenced by hardness value using linear Bailey-Hirsch relationship, as shown in Eq. 1 [7].

$$HV\ (GPa) = 0.7 + 1.5Gb\rho^{1/2} \qquad \text{(Eq. 1)}$$

where HV is Vicker hardness measured in 316L (GPa), G is iron shear modulus of 80 GPa and b is burger vector of FCC structures of austenitic stainless steel which is 2.49Å.

Experimental Procedure

Austenitic stainless steel 316L implant plates utilized in this experiment was supplied by PT Pelopor Teknologi Implantindo, Mojokerto, Indonesia. The sample was solution-annealed at temperature of 1150 °C within 1 hour and quenched in water after casting process in order to homogenize their microstructures. The thickness was 4.3 mm with a length of 200 mm. SpectroMax Optical Emission Spectroscopy (Ametex, Germany) reveals the chemical composition of 316L as listed in Table 1. The investment casting 316L implant plate undergoes 20 passes of unidirectional cold rolling until approximately 50% reduction in thickness is attained as shown in Fig. 1. The thickness was reduced from 4.3 mm to 2.05 mm.

A b

Fig. 1. (a) As-cast implant plate 316L and (b) implant plate 316L after 50% reduction by cold rolling

Table 1. Chemical composition of austenitic stainless steel 316L

Chemical Composition									
C	Cr	Ni	Mo	Mn	P	S	N	Cu	Fe
0.01	15.79	13.51	3.71	0.91	0.02	0.005	0.124	0.12	65.10

The cold-rolled sample was cut into 30 x 18 mm and then thermally cycled in different temperatures of 850° C, 900° C and 950° C with holding time of 35 seconds and repeated within four cycles. Fig. 2 illustrate the process employed in this experiment. All of the samples including the investment-casting and the cold-rolled were grind and polished stepwise using SiC grit paper prior to microstructure examination using Olympus Stereo Microscope and SEM Hitachi FlexSEM 1000. The average grain size measurement was calculated based on the standard linear intercept method according to ASTM E 112. Microvickers hardness testing was employed according to ASTM E92 taken at 10 different points to generate average hardness value using a load of 1 Newton with a dwell time of 10 s.

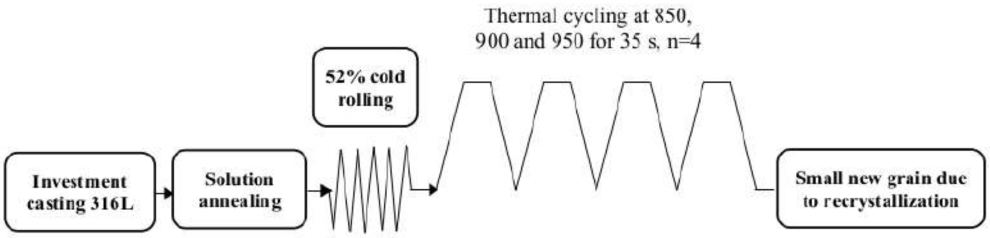

Fig. 2. Schematic process employed during the experiments.

Result and Discussion

Dislocation density

Based on the actual measured thickness, 52% of thickness reduction was achieved after several steps of cold-rolling process. Eq. 1 deliver the dislocation density based on measured hardness at 52% cold reduction of 407 $HV_{0.3}$ (3.98 GPa) with a value of dislocation density of 1.22×10^{16} m^{-2}.

The microstructural changes due to cold-rolling process were presented in Fig 3. The investment casting produces dendritic microstructure as seen in Fig 3a. The cold rolling at 52% of thickness reduction reveals angular grain of medium sphericity not the elongated grain such as mostly detected in cold rolling of steel in Fig 3b. The average grain size measurement provides 290 μm and 198 μm values of the investment casting and cold rolled at 52% in thickness reduction samples respectively.

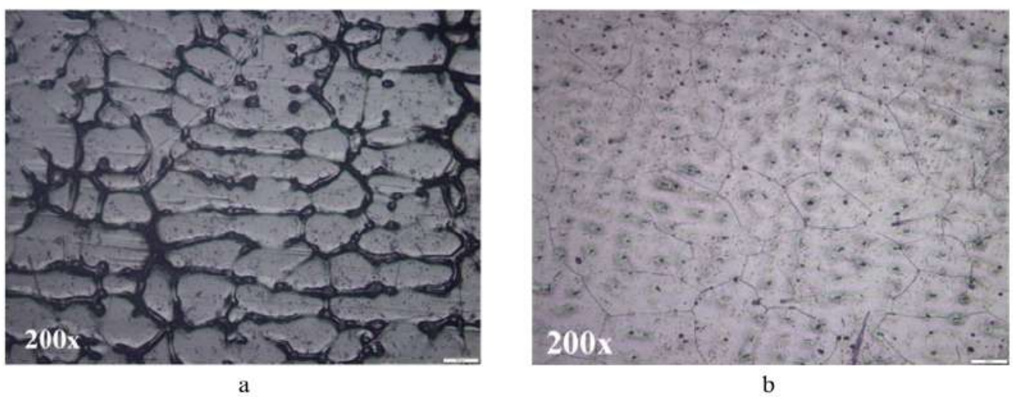

Fig. 3. (a) As-cast 316L dendritic microstructures and (b) after cold-rolling with 52% in thickness reduction

The hardness value for each sample tested during these experiments was shown in Fig. 4. The investment casting sample only had hardness value of 155 $HV_{0.3}$ and the value increased to 407 $HV_{0.3}$ after being cold-rolled to 52% reduction in thickness. The high hardness indicates that the material has substantial residual stress or stored strain energy generated after several passes of cold-rolling process. After thermal cycling treatment, the hardness had decreased as the stored strain energy is gradually being released due to elevated temperature exposures. Thermal cycling at 850 °C and 900 °C slightly decrease the hardness values indicating that only small amount of stored strain energy is being released. This condition was not enough to reach recrystallization kinetic to allow nucleation of new grain. Higher thermal cycling temperature at 950 °C had resulted in lower hardness as a massive amount of stored strain stress is being released and accelerating recrystallization process by which deformed grains are consumed and nucleation of new finer grain was taking places. Thermal cycling at 950 °C reduces the hardness to 253 $HV_{0.3}$ which is 38% lower as compared to the cold rolling at 52% thickness reduction.

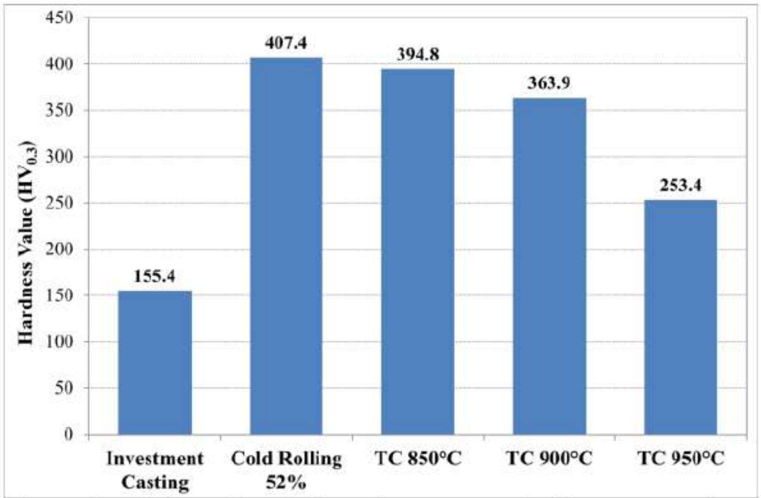

Fig. 4. The hardness value of thermally cycled samples as compared to investment casting and cold-rolled samples.

The microstructures evaluation of thermally cycled samples at 850, 900 and 950 °C presented in Fig. 5 reveals that only after exposure to 950 °C that the grain becomes much finer with an angular shape. This condition can only happen if the recrystallization process had taken place as the stored strain energy was massively relieved aiding nucleation of new grain from the grain boundary of deformed microstructures. By limiting the exposure time to 35 seconds, there were not enough energy available to allow grain growth thus keeping the grains in finer size. The number of cycles was meant to allow the nucleation of the new grain in each cycle process until all the deformed grain was fully converted.

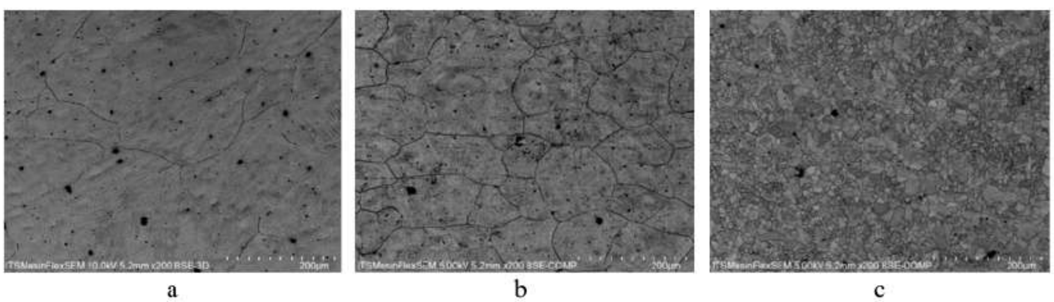

Fig. 5. Microstructures of thermally cycled samples after 4 cycles at (a) 850 °C for 35 seconds (b) 900 °C for 35 seconds and (c) 950 °C for 35 seconds. Note that the recrystallization only happens at 950 °C thermal cycling.

The calculated grain size measurement of thermal cycling at 850 °C and 900 °C was providing value rather similar to cold rolling at 52% reduction condition. This was in accordance with the hardness measurement results in Fig. 4. Based on the microstructures in Fig. 5, the finer grain size of 22 μm on average was calculated after thermally cycled at 950 °C. This value is more than ten times smaller as compared to the investment casting grain size of 290 μm on average.

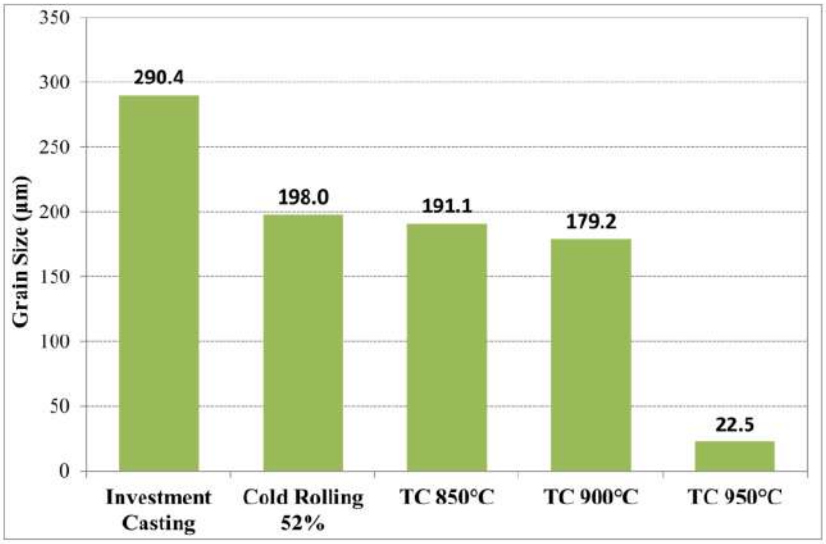

Fig. 6. The average grain size of thermally cycled samples at different temperatures exposures as compared to investment casting and cold-rolled samples.

Fig. 6 shows the comparison of calculated grain size of thermally cycled samples at different temperatures exposures with investment casting and cold-rolled samples grain size. The recrystallization process that led to finer grain size only happen after thermally cycled at 950 °C. This thermal cycling temperature was found to be higher as compared to Nanda [6] finding. In their experiments, the recrystalization has mostly occurred at 900 °C thermal cycling temperatures regardless of the amount of cold rolling reduction [6].

Conclusion

Thermal cycling treatment at 950 °C following cold rolling at 52% thickness reduction had resulted in recrystallization mechanism that initiates nucleation of a new finer grain of 22 µm as compared to investment casting grain of 290 µm size and cold rolling 52% grain of 198 µm in size. The decrease in hardness after thermal cycling exposure indicates that stored strain energy generated during cold-rolling process was gradually dispersed. Unfortunately, lower thermal cycling temperature than 950 °C only able to slightly relieved the strain and not able to initiate nucleation of new finer grain led to recrystallization.

Acknowledgment

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