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Recovery and Recrystallization Behaviour of Super 304h-Cu Austenitic Stainless Steel

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ABSTRACT: SS 304 H is a fully austenitic stainless steel having fcc crystal structure from room temperature to melting point and does not involve any solid state phase change up to melting. Hence their microstructure and mechanical properties cannot be modified by transformation induced by heat treatment unlike in the case of ferritic steels. Hence the only way to modify its property and microstructure is by hot or cold rolling, followed subsequently by suitable annealing. Hence a basic understanding of strain energy relaxation in the process of recovery, refry stylization and grain growth is essential in this particular material.

I. INTRODUCTION

In this paper the results obtained through different characterization techniques have been presented. This is followed by a detailed discussion on the strain energy relaxation in the processes of recovery and recrystallization respectively. The detailed discussion on the present results are based on both quantification of stored energy variation using DSC and microhardness measurement and the subsequent microstructure evolution by the process of recovery and recrystallization in plastically deformed 304H SS upon thermal annealing both in isothermal and nonisothermal conditions.

II. OPTICAL MICROSTRUCTURE OF FULL ANNEALED AND COLD ROLLED SS 304H

The optical microstructure of the SS304H sample annealed at 1200°C for 2 hour/water quenched to room temperature is shown. In **figure**. The microstructure shows uniform equiaxed grains with average grain size of 32 μ m.Appearance of Annealing twins reveals the characteristics of annealed austenitic steel. The micro hardness value is found to be around 175 VHN. It also shows that 1200 °C annealing could not dissolve all the primary NbCN phase, which are seen as black thick particles. However, most of M₂₃C₆ type carbides have already seen full dissolution at the end of 1200 °C/2h treatment.

This annealed sample of SS 304H with a initial dimension of 11cm x 4cm x 3cm was subsequently cold rolled at room temperature to 80% thickness reduction. In **figure.** The optical micrograph of 80% cold rolled microstructure is shown. The grains are considerably elongated in the rolling direction with sharp and irregular boundaries as indicated by the arrow mark. In general it is noticed that the deformation is highly inhomogeneous as it is expected in in case of severely

deformed austenitic stainless steels . The average microhardness of the deformed microstructure is found to be 510 VHN for 80% cold worked conditions, where the litreature value reported for 316 SS is about 502 VHN .



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Fig. Room temperature

In order to study the rate of lattice softening with respect to temperature in the process of recovery, recrystallization and grain growth, constant time annealing treatment have been carried at different temperatures. The annealing treatments were performed in a muffle furnace at various temperatures in the range 550°C (823K) – 950°C (1223K) and for time durations varying from 1 minute to 5h followed by water quenching to retain the microstructural aspects. These samples have been characterized by standard metallographic and hardness techniques to obtain the extent of recovery and recrystallization as a function of both temperature and time. Figure 4.4 shows the variation of microhardness with temperature of annealing at four different time of holding. The typical nature of the curves



Fig.Non-Isothermal hardness variation at various fixed holding

represent different stages of kinetics involved in different processes such as recovery, recrystallization and grain growth. These are marked clearly in the figure For a particular time of holding the slope of the hardness curve varies very slowly with temperature, up to $0.6T_m$ (~920K). This is partly because of the annihilation of point defects and a local rearrangement of dislocations towards a low energy state. This particular stage where the variation of dislocation density is found to be slow and somewhat steady is known as recovery.

In the next stage, above 920 K and up to 1050 K, the hardness value falls rapidly for all times of holding in a nearly identical fashion. This region is where recrystallization is the dominant mechanism. The sudden fall in hardness confirms the nucleation of new strain free grains in recrystallization. These newly formed grains then start to grow to minimize the grain boundary energy and this particular stage is often referred to as grain growth. This fact has also been observed from the metallographic studies which are to be reported later in this chapter. Different temperature domains for recovery, recrystallization and grain growth has approximately marked in figure. It is also clear from the figure that the effect of holding time on hardness variation at a particular temperature follow a definite trend and hence



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it is possible to quantify the variation of dislocation density and stored energy variation as a function of temperature and time.



Variation of microhardness with time



The time, temperature variation of hardness data has been analysed in the following section. Variation of Hardness upon Isothermal annealing treatment for 80% cold work super 304H Cu



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Fig.Non-Isothermal hardness variation at various fixed holding

Figure shows the change in hardness with respect to time for 80 % cold work alloy at different temperature. It is clearly evident that at each temperature, the hardness value reaches to a low saturation value after the lapse of certain time. The time after which the microhardness starts to saturate is found to be different for different temperature processes. In fact it is low for high temperatures. In addition, it is also clear from figure. that the variation of hardness with time at low temperature has two distinct regions. This is because, at relatively low temperatures recovery process is the dominant mechanism. Recrystallization is yet to assert in a major way.

The time variation of hardness has been converted to extent of lattice softening versus time by using following conversion formula given in Eq.

Fig. Fraction of recrystallization as function of time for different Temperatures

Where H(t) is hardness at any time t, H_{ann} is the hardness of fully annealed sample at a maximum annealing time and H_{cw} is the hardness of starting 80% coldwork sample. The plot of fraction softened as a function of time is presented in figure. From the above experimental result one cannot clearly distinguish between recovery and recrystallization as they have overlapping occurrence. It is possible to separate the influence of recovery by following a theoretical procedure suggested by Stuwe .



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Fig.Optical Micrograph super 304H annealed



Fig:Optical Micrograph for80% cold



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III. MICROSTRUCTURE EVOLUTION UPON ISOTHERMAL & NON-ISOTHERMAL ANNEALING OF SS304H CU

The changes in microstructure with different annealing temperature for a fixed annealing time of 15 min are illustrated. It can be clearly identified from the figure that ata temperature of 800°C nucleation of new strain free grains appears which indicates the onset of recrystallization though in some regions the presence of deformed grain boundaries can also be seen. A fully recrystallized microstructure is only found for the annealed sample at 900 °C with no sign of deformed grains. This observation well agrees with our calorimetry results that will be discussed in the next section. The microstructures for samples bellow 800°C shows clearly the deformed



Fig 4.9: Effect of time on the recrystallization microstructure of 80% cold rolled SS304H-Cu that is annealed at 800°C(the microhardness values are indicated at the top right)

grains with no indication of nucleation which revealed that microstructural morphology does not change significantly up to temperature 800°C and this is also supported by hardness measurements in this temperature zone.

Since it has already been discussed that the process of lattice softening by recovery & recrystallization depends both on time and temperature, the time evolution of microstructure has also significant fundamental importance to study the kinetics of the process. A microstructural collage for several samples annealed at 800°C at different time of

annealing is shown in figure. It is clearly evident from the figure that for 1 min and 5 min of annealing there is no significant recrystallization took place whereas for 15 minutes of annealing freshly nucleated strain free grains are observed. There is a gradual disappearance of parent deformed grain boundaries and at 2 hrs of annealing the material is seen to be fully recrystallized with no microstructural feature of a deformed material. Hence only for annealing time above 2 hrs at temperature 800°C, SS304H becomes fully recrystallized. These strain free grains further found to grow in the third stage of relaxation process called grain growth. The process of grain growth leads to reduce the no of grains in the material, where relatively larger grains start to grow with the expense of smaller grains. This minimizes the grain



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boundary energy per unit volume of the material. This can be seen from the microstructure for 5hr annealed sample where the average grain size becomes $10 \ \mu m$.

IV. CONCLUSION

In this study, Recrystallization behaviour of super 304H-Cu austenitic stainless steel has been studied using dynamic calorimetry technique with metallography and hardness measurement as supplementing contributions. The summary of the present work is as follows. Theoptical microstructure of annealed SS304H showed equiaxed grains with the copious presence of annealing twins similar to that of typical austenitic stainless steel. The microhardness value for the annealed sample is found to be around 175 VHN. Whereas the average microhardness of the deformed microstructure is found to be 510 VHN for 80% cold worked conditions. From the microstructural characterization of isothermally and isochronally annealed samples, it is obvious that the effect of temperature and time both has a significant impact on the recrystallization behavior of SS304H Cu.

REFERENCES

- 1. William D.Callister, Jr, *Material Science and Engineering an Introduction*, Wiley Student edition, sixth edition.
- 2. R.E. Smallman and A.H.W. Ngan, *Physical Metallurgy and Advanced Materials*, Elsevier Ltd, Seventh edition 2007.
- 3. V. Raghavan, Materials Science and Engineering, Prentice Hall of India, New Delhi, Second edition ().
- 4. Sidney H. Avner, Introduction to Physical Metallurgy, Tata McGraw Hill, New Delhi, Second edition
- 5. R. Visawnathan, W. Bakker, Materials for ultra supercritical coal power plants boiler materials: Part 1, JMEPEG 10 (2001) 81–95
- 6. T. Kan, Y. Sawaragi, Y. Yamadera, H. Okada, Proceedings of 6th Liege Conference on *Materials for Advanced Power Engineering*, Part-1, 1998, pp. 441–450
- 7. Akira Tohyama, Hitoshi Hayakawa, and Yusuke Minami- NKK technical review No. 84(2001)
- 8. Minami et al. Iron and Steel. Vol. 71, No. 13, pp. S1447 (1985).
- 9. F.J. Humphreys and M. Hatherly, Recrystallization and Related Annealing Phenomena, Pergamon Press, Oxford, 1995.
- 10. P. Cotterill and P.R. Mould, *Recrystallization and Grain Growth in Metals*, Surrey University press, London.