Non-Destructive Evaluation of Embrittlement in Isochronal Aged Fe-Cr Alloy by EMAR Technique

EMAR 法による等時焼鈍した Fe-Cr 合金の脆化の非破壊評価

J. N. Mohapatra^{1‡*}, Y. Kamada¹, J. Echigoya¹, T. Ohtani², D. G. Park³, H. K. Jung³ and Y. M. Cheong³ (¹NDE and Sci. Res. Center, Faculty of Eng., Iwate Univ.; ²Shounan Inst. of Tech.; ³Korea Atomic Energy Res. Inst.)

J. N. Mohapatra^{1‡*}, 鎌田康寛¹, 越後谷淳一¹, 大谷俊博², D. G. Park³, H. K. Jung³ and Y. M. Cheong³ (¹岩手大 工;²湘南工科大;³韓国原研)

1. Introduction

Fe-Cr alloys are important engineering materials for the components of nuclear and chemical industries ¹). Embrittlement in such alloys arises due to the formation of Cr rich α' phase or σ phase depending on the Cr content and heat treatment conditions affects largely to the mechanical properties ²⁻⁴. Therefore, there is a need for the development of suitable Non-Destructive Evaluation (NDE) techniques, for the inspection of in-service components for its safe operation. Most of the previous studies using ultrasonic method were carried out using conventional technique which inherently includes contact problems. In order to have a precise and reliable measurement, the data scattering by contact problem should be electromagnetic avoided. Therefore, acoustic resonance (EMAR) method, a contact less measurement, attracts much attention for the characterization of material in a fine scale 5-8. In the present investigation, Fe-Cr binary alloy was isochronal aged and inspected by EMAR method.

2. Experimental Procedure

Fe-48% Cr alloy was prepared in arc melting technique. The alloy was solution annealed at 1000 ^oC for 2 hr and then water quenched. Specimen $(L_1$ \times L₂ \times L₃= 7.0 mm \times 2.0 mm \times 5.0 mm) was cut from the alloy for EMAR measurement in the frequency range from 0.25 to 1.5 MHz. Fig. 1 shows the schematic representation of RITEC-RAM system and specimen configuration. The details of measurement procedure can be found elsewhere ⁷⁾. Fig. 2 shows the typical spectrum and ring-down curves measured by EMAR. The ring-down curve was approximated by the exponential function to determine the attenuation coefficient α . Isochronal ageing was carried out to the specimen by lamp heater in high vacuum in the temperature range from 400 to 700 °C with 50 °C step. At each ageing stage, the holding time was

1hour. The hardness measurement was performed by an automatic micro Vickers hardness tester.



Fig. 1 Experimental setup for EMAR measurement



Fig. 2 Typical spectrum and ring-down curves measured by EMAR.

3. Results and Discussion

Fig. 3(a)-(d) shows the change in hardness, resonant frequency, volume of the specimen and attenuation coefficient with isochronal ageing. The resonant frequency and volume were normalized by their initial values.

jnmohapatra@gmail.com

Fig. 3(a) shows that the hardness of alloy increased with isochronal ageing up to 500 °C and then decreased. The increase in hardness is due to the formation of coherent Cr rich α' phase which arises in the material by Fe-Cr phase separation. The increase in Cr rich α' phase enhances the coherent stress in the alloy and hence the hardness of the alloy was increased. Above 500 °C, the coherent stress disappears and hence the hardness of the alloy decreased. Such results well correlate with the available literature ¹⁾. It has been reported that the hardness increases at 700 °C due to the formation of σ phase ¹⁾. No increase in hardness at 700 °C indicates that the σ phase was not produced in the present specimen probably due to the short ageing time ⁹⁾.

Fig. 3(b) shows the change in resonant frequency with ageing. The resonant frequency increased similar to the change in hardness in the alloy up to 500 °C and then decreased. The resonant frequency is related to both the elastic constant and the specimen size. Fig. 3(c) shows the volume change calculated from the specimen size measured by micrometer. Since the volume change did not occur with ageing, the α - α' phase separation in Fe-Cr alloy resulted in the increase of the elastic constant of the alloy ⁸⁾. Therefore the resonant frequency of the alloy increased up to 500 °C.



Fig. 3 Ageing temperature dependence of Vickers hardness, resonant frequency, volume and attenuation coefficient of the specimen.

Fig. 3(d) shows the change in attenuation coefficient with ageing. The result shows that the attenuation coefficient decreased at the first annealing at 400 $^{\circ}$ C, whereas no further change has been observed with increase in isochronal ageing temperature.

4. Conclusion

EMAR measurement was carried out in isochronal aged Fe-48% Cr alloy. The resonant frequency of the alloy increased up to 500 °C and then decreased corresponding to the change in hardness. A good correlation is found between the resonant frequency and hardness indicating the feasibility of EMAR technique for the evaluation of thermal embrittlement in Fe-Cr alloys.

Acknowledgement

This work was supported in part by Japan Society for the Promotion of Science and Korea Science and Engineering Foundation under the contract of the Japan-Korea Basic Scientific Cooperation program.

References

- 1.Y. Yustinovshikov, M. Shirobokova and B. Pushkarev: Acta Mater. 44 (1996) 5021.
- 2.Y. De Carlan, A. Alamo, M. H. Mathon, G. Geoffroy, A. Castaing: J. Nucl. Mater. 672 (2000) 283.
- 3. M. K. Miller, J. M. Hyde, M. G. Hetherington, A.Cerezo, G. D. W. Smith and C. M. Elliott: Acta Metal. Mater. **43** (1995) 3385.
- M. K. Miller, J. M. Hyde, A. Cerezo, G. D. W. Smith: Appl. Surface Sci. 87/88 (1995) 323.
- M. Hirao and H. Ogi, *EMATs for Science and Industry*, (Kluwar Academic Publishers, Boston, 2003)
- T. Ohtani, F Yin and Y. Kamada: Jpn. J. Appl. Phys., 47, (2008) 3916.
- Y. Kamada, S. Chigusa, T. Othani, H. Kikuchi and S.Kobayashi: Proc. USE. 29 (2008) 49.
- 8. M. Tane, T. Ichitsubo, H. Ogi, M. Hirao: Scripta Mater. **48** (2003) 229.
- A. Blachowski, S. M. Dubiel, J. Zukrowski: J. Cieslak, B. Sepiol: J. of Alloys and Comp. 313 (2000) 182.