INFLUENCE OF AGE-TREATED FINE-SCALE MICROSTRUCTURE ON THE FRACTURE BEHAVIOUR OF INCONEL X-750

A. K. Sinha, M. G. Hebsur and J. J. Moore

Mineral Resources Research Center, University of Minnesota, Minneapolis, MN 55455, USA

ABSTRACT

Age-hardening nickel-base alloy Inconel X-750, which is used extensively in PWR and BWR components, has exhibited intergranular fracture under service conditions due to improper heat treatment. At present there seems to be no recognized standard aging treatment for this alloy, but the usual process is to use a triple heat treatment which comprises solution treatment at 1149°C/2 hours, air cool, + 843°C/24 hours, air cool, + 704°C/20 hours, air cool. The present study investigates the influence on the fracture behaviour of varying microstructural features produced by single aging between 704-871°C after solution annealing at 1075°C for two hours.

Inconel X-750 obtained by electroslag refining has been subjected to single aging treatments ranging from 704 to 871°C for times of 2 hours to 200 hours. The fine-scale microstructures were well documented using optical microscopy, TEM and SEM. Fracture studies were carried out using notched charpy specimens given the treatment mentioned above and the fracture surfaces were examined using scanning electron microscopy.

KEYWORDS

Single aging treatment; apparent fracture toughness; CVN absorbed energy; tearing; quasi-cleavage fracture; intergranular and ductile fracture mechanisms.

INTRODUCTION

Inconel X-750 is a high strength, age hardenable nickel-base superalloy with good oxidation and corrosion resistance (Inconel Alloy X-750, 1979; Kattus, 1981; Raymond; 1967). As a result of these properties, this alloy has been widely used in both BWR and PWR applications as bolts, springs, pins and other structural hardware components (Boisde and co-workers, 1971; Mills, 1981).

This austenitic alloy constitutes mainly the γ matrix, precipitation of "intermetallic" $\gamma'(Ni_3Al$ or Ni_3 Ti) within the grain; the carbides such as MC type (Ti,NbC), and $M_{23}C_6$ carbides (e.g., $Cr_{23}C_6$). The formation of the $M_{23}C_6$ type is believed to occur by the following reaction:

$$MC + \gamma \rightarrow M_{23}C_6 + \gamma'$$

The present study was undertaken with the view to assess the influence of varying microstructural features produced by single aging (Hattori, and co-workers) between 704 and 871°C on the fracture behaviour. In this temperature regime most of the precipitation sequences occur rapidly.

Optical and transmission and scanning electron microscopy were used in the present study.

EXPERIMENTAL PROCEDURE

The chemical composition of the electroslag refined heat used in this study is given in Table 1. Samples were cut from a 1.5-inch (38 mm) diameter rod

TABLE 1 Chemical Composition (by Weight %)

Elements										
C	Mn	Fe	S	Si	Cu	Ni	Cr	Al	Ti	Nb+Ta
0.06	0.19	7.82	0.001	0.40	0.37	72.02	14.82	0.85	2.49	0.98

and were given various heat treatment cycles comprising solutioning treatment at 1075°C for 2 hours, followed by water quenching. Subsequently, aging treatments at 704°C, 760°C, and 871°C, between 2 and 200 hours followed by air cooling were conducted. This aging temperature range was selected to facilitate a better understanding of the entire precipitation sequence (Mager and Aspden; private communication with Huntington Alloys, Inc., 1983).

Metallographic specimens were prepared for optical microscopy (Sinha, Hebsur and Moore, 1983). The microstructural changes occurring during aging were documented on thin foil specimens examined under the transmission electron microscope at an accelerating potential of 100 kV. Thin foils were prepared by electrochemical thinning at room temperature in a bath containing 10% perchloric acid in butanol and under a potential of 50 V. Energy dispersive X-ray (EDX) analyses with SEM and SAM point analyses were taken on polished specimens in order to determine the primary (MC type) and secondary ($M_{23}C_6$ type) carbides present within the grain and along the grain boundaries of the heat-treated samples (Sinha, Hebsur and Moore, 1983).

ASTM standard charpy impact test specimens were machined for each heat treatment cycle in the longitudinal forming direction. Specimens were subsequently tested on the Dynatup Instrumented Impact Machine in air.

Apparent fracture toughness (Server, Ireland, and Wullaert, 1974) and CVN values were determined from instrumental impact data. The fracture surfaces were examined under a Philips 500 scanning electron microscope at an

accelerating potential of 12.5 kV.

RESULTS

Figure 1 shows the aging curve which indicates that aging at 704°C, 760°C,

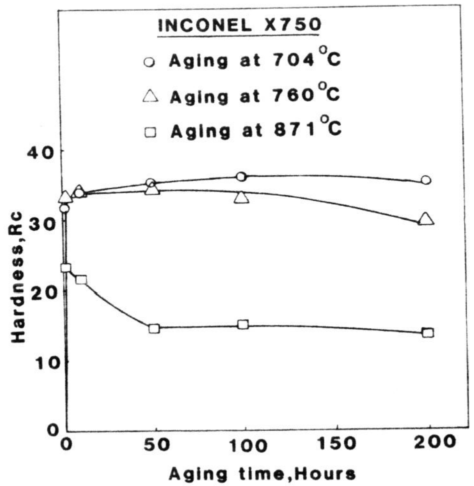


Fig. 1. Hardness versus aging time.

and 871°C, each followed by air cooling, results in a peak hardness at 100 hours, 50 hours and 2 hours, respectively. Figure 2 illustrates the typical microstructures of heat treated specimens aged for 2 and 200 hours at 871°C.

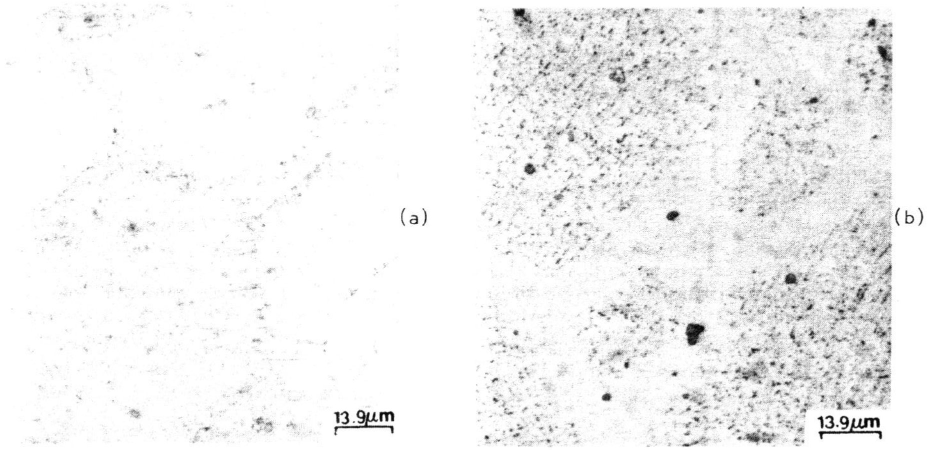
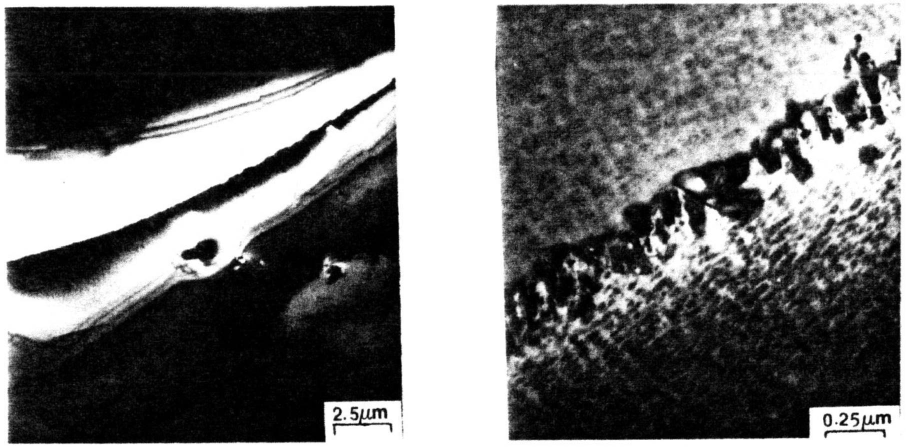


Fig. 2. Inconel X-750 solution treated, water quenched and aged at (a) 871°C, 2 hrs. and (b) 871°C, 200 hrs.

Transmission electron photomicrographs (Fig. 3) of the grain boundary region illustrate continuous carbide precipitation after aging for 2 hours at 704°C (Fig. 3(a)), which breaks up into discrete and discontinuous particles after aging at 704°C for 100 hours. The intermetallic phase γ' (Ni_3Al, Ni_3Ti) is evident in the grain interior in Fig. 3(b), together with a small zone adjacent to the grain boundary which is depleted in chromium. It was found that these continuous carbides began to break up into discrete and discontinuous particles after 50 hours aging at 704°C and 760°C (Sinha, Hebsur, and Moore, 1983). It is thought that these carbides are predominantly $M_{23}C_6$, e.g., $Cr_{23}C_6$ with some MC type carbides, e.g., NbC, TiC, also present after prolonged aging. Further work is continuing to provide a more complete characterization of the chemistry of these grain boundary carbides for these single aging treatments.



(a) (b)

Fig. 3. Typical TEM microstructures corresponding to aging at 704°C for (a) 2 hrs. and (b) 100 hrs. aging times.

Figure 4 shows the room temperature CVN absorbed energy and apparent fracture toughness of Inconel X-750 as a function of aging time. This indicates that both the apparent fracture toughness and the CVN values decrease with aging time for 704°C and 760°C aging temperatures. On the other hand, both these values increase with aging time for specimens aged at 871°C. However, the highest CVN absorbed energy and apparent fracture toughness values have been observed for the specimens solution treated at 1075°C for 2 hours followed by water quenching, presumably due to the ductile matrix in the supersaturated solid solution. SEM fractographs, obtained on broken charpy specimens, are illustated in Figs. 5-7.

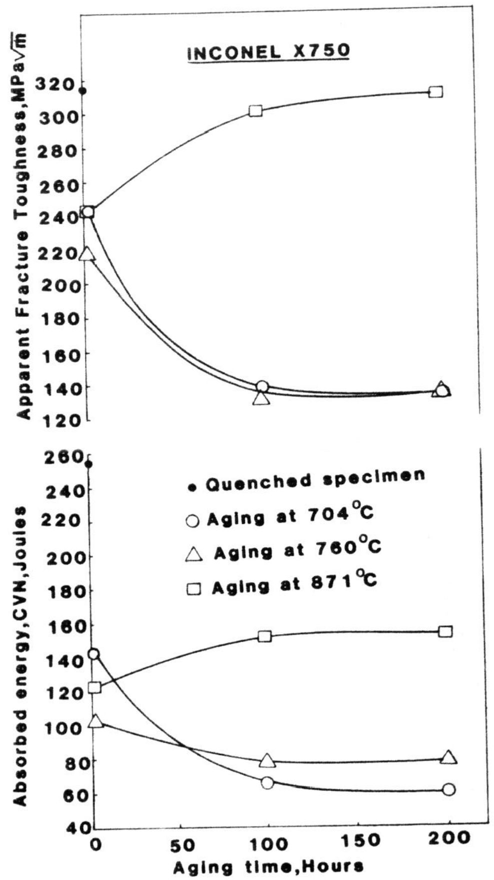


Fig. 4. Effect of aging time on impact properties of age treated Inconel X-750.

DISCUSSION

The maximum peak hardness values are obtained as a result of the strengthening effects of the coherent intermetallic precipitates, γ'. The formation and growth of incoherent intermetallic precipitates cause reduction in the hardness values beyond the peak points. Since the solution temperature of Cr-rich $M_{23}C_6$-type carbides is about 871°C (Inconel Alloy X-750, 1979), aging at 871°C followed by air cooling causes the dissolution of this carbide and finally a secondary carbide-free zone is established throughout the matrix after aging for 200 hours at 871°C (Fig. 2(b)) (Sinha, Hebsur, and Moore, 1983).

The continuous grain boundary $M_{23}C_6$-type carbides which are present at the

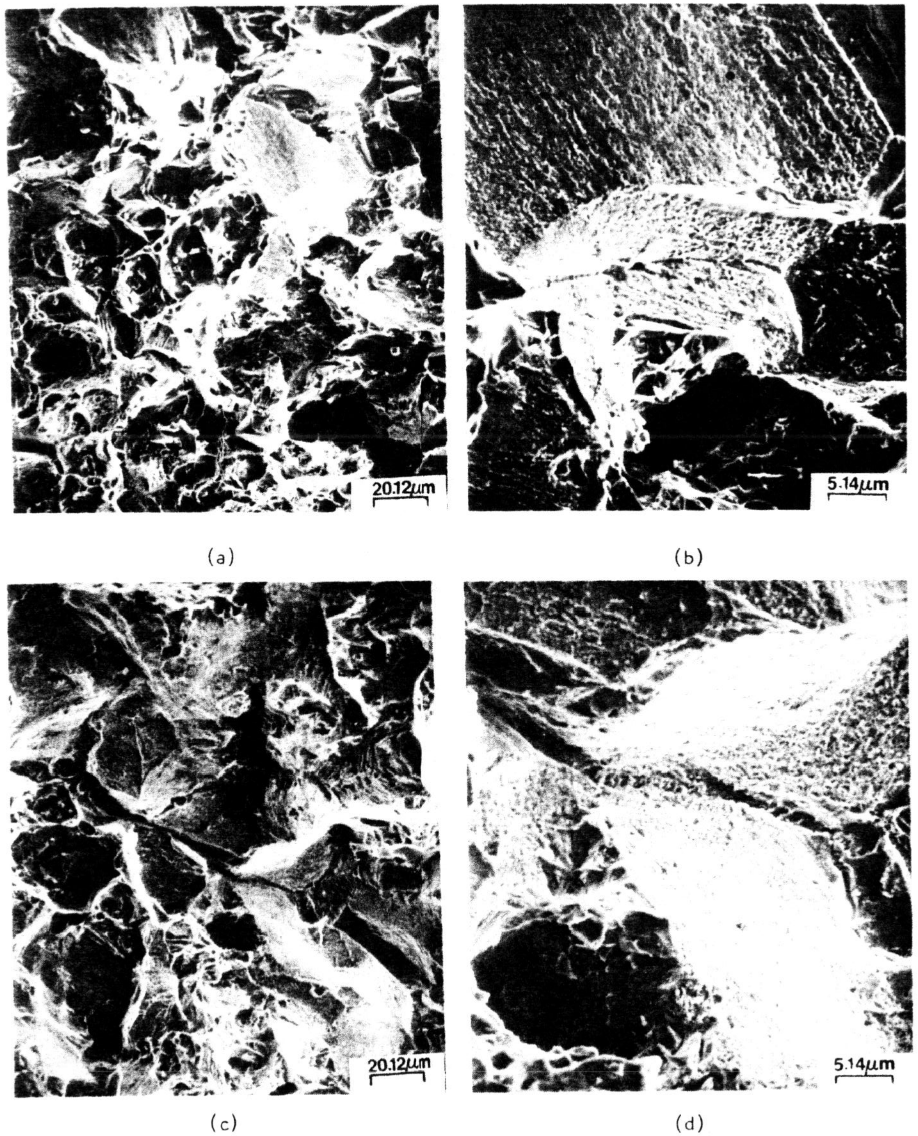


Fig. 5. SEM fractographs of impact specimens aged at (a,b) 704°C for 100 hrs. and (c,d) 704°C for 200 hrs. at different magnifications.

lower aging times begin to break up and become discrete after 50 hours aging at 704°C and 760°C. As time progresses beyond 50 hours, Cr replenishment of the grain boundary occurs and grain boundary carbides are subjected to

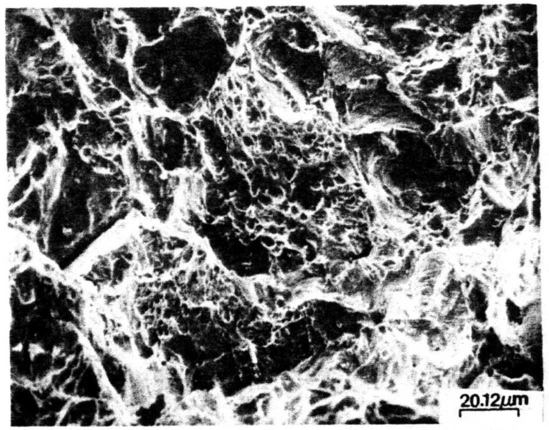


Fig. 6. SEM fractograph of impact specimen aged at 760°C for 2 hrs.

restructuring to form more discrete particles (Sinha, Hebsur, and Moore, 1983).

The fracture surfaces were examined in order to relate impact properties and hardness to operative fracture mechanism(s). Aging at 704°C for 100 and 200 hours, the Inconel X-750 fracture surfaces exhibited predominantly intergranular fracture; some transgranular faceting and a few cleavage facets were also visible (Fig. 5). The rumpled appearance observed on the intergranular facets was attributed to a dimple rupture network. In this alloy, this intergranular fracture behaviour is believed (Mills, 1980) to result from microvoid coalescence along the grain boundary denuded zone (Fig. 3). It appears that extensive plastic flow around the grain boundary carbides results in a stress concentration at carbide-matrix interface which leads to a decohesion between the carbide particle and the matrix. Fig. 5 (b,d) illustrates the small spherical particles within cavities or dimples, believed to be $M_{23}C_6$ carbides, that initiated the microvoids. Subsequent growth and coalescence of these microvoids within the grain boundary denuded zone produces intergranular dimple rupture network (Mills, 1981; Mills, 1980). At the intermediate aging temperature (760°C) the surface exhibited a limited amount of intergranular fracture coupled with mostly shear or elongated dimples and some tearing ridges and regions (Fig. 6). At a higher aging temperature (871°C) hardness decreases appreciably and consequently impact properties increase with aging time; the secondary carbide, e.g., $M_{23}C_6$, along the grain boundary starts dissolving after 2 hours and is replaced entirely by a secondary carbide free zone after 200 hours aging time (Fig. 2(b)). Hence fracture surfaces at this aging temperature for 2 hours show very few intergranular fracture appearances and at 100 and 200 hours exhibit the transgranular ductile fracture (Fig. 7). At this aging temperature, dimple size appears to increase with aging time.

All these fracture surfaces mentioned above are also associated with the breaking of intermediate and large cubic shaped MC-type carbides, i.e., NbC, TiC, within very large dimples causing microvoid formation and subsequent growth of voids leading to quasi-cleavage fracture (i.e., involving both microvoid coalescence and cleavage). It is anticipated that quasi-cleavage

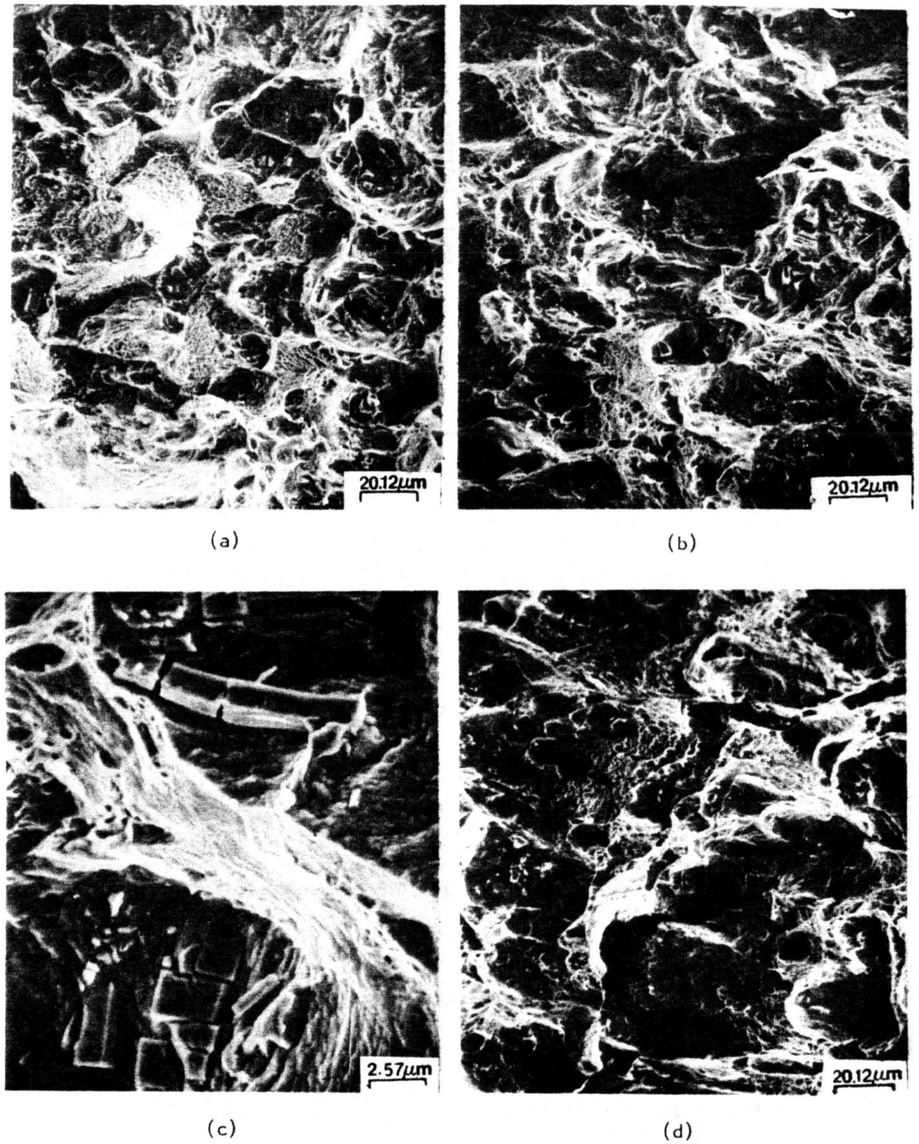


Fig. 7. SEM fractographs of impact specimens aged at (a) 871°C for 2 hrs., (b,c) 871°C for 100 hrs. at different magnifications, and (d) 871°C for 200 hrs.

facets fracture ahead of the moving crack front; then, with the increase in stress, the cleavage facet extends by tearing into the matrix around it by

microvoid coalescence (Metals Handbook, 1974). A tearing region is invariably observed adjacent to the large dimples or between the two large dimples containing broken MC-type carbides. Tearing occurs if the ductility is sufficiently high and the flow stress sufficiently low (Metals Handbook, 1974). It appears that voids do not coalesce with each other, but are individually intersected by and coalesced with the tip of a growing crack (Rao and Alexander, 1983).

CONCLUSIONS

Operative fracture mechanisms have been related to the impact behaviour, microstructure, and hardness of the precipitation heat treated Inconel X-750 solution treated and aged between 704 and 871°C. The following conclusions can be drawn.

(1) Inconel X-750 alloy exhibited a maximum hardness in the samples aged at 704°C for 100 hours, 760°C for 50 hours, and at 871°C for 2 hours, followed by air cooling. A complete secondary carbide free zone was present after aging at 871°C for 200 hours.

(2) Apparent fracture toughness and CVN values decrease with aging time at 704°C and 760°C aging temperatures, while these values increase with aging time for specimens aged at 871°C.

(3) Aging at 704°C for 100 and 200 hours (coincident with lower apparent fracture toughness and CVN values and higher hardness values), the fracture surfaces exhibited mainly intergranular fracture coupled with some transgranular faceting and a few cleavage facets. The fracture occurred by an intergranular dimple-rupture mechanism attributed to microvoid coalescence around the grain boundary carbide.

(4) An improved fracture resistance was produced on aging at 871°C and was thought to result from a transition from partial intergranular to a complete ductile (transgranular) fracture mechanism. The dimple size appeared to increase and the larger dimples were more numerous with aging time at this temperature.

(5) A tearing region was invariably observed adjacent to the large dimples and between the two large dimples containing broken MC-type carbides.

(6) A predominance of large dimples containing broken intermediate and large cubic shaped MC-type carbides was observed on the fracture surface of specimens aged at 871°C compared with those aged at 704°C and 760°C.

AKNOWLEDGEMENTS

The authors wish to express their appreciation to Mr. Tony Matuszewski, Materials Division, Harnischfeger, Inc., Milwaukee, Wisconsin, for help in Instrumental Impact Testings. Financial support for this project was provided by the Department of Energy through the Corrosion Research Center of the University of Minnesota under Contract DOE/DE-AC02-79ER/0450.

REFERENCES

Boisde, G., H. Cariou, L. Grall, and R. Mahut (1971). Effects of Environment on Material Properties in Nuclear Systems. The Institution of Civil Engineers, International Conference on Corrosion, London. pp. 111-118.

Fractography and atlas of fractographs (1974). Metals Handbook, Vol. 9, 8th ed. Metals Park, Ohio. pp. 64-92.

Hattori, S., Y. Mori, L. Masaoka, H. Itoh and R. Watanabe. Hitachi Research Laboratory, Hitachi, Ltd.

Inconel Alloy X-750 (1979), 4th ed. Huntington Alloys, Inc.

Kattus, J. R. (1981). Aerospace Materials Handbook, Vol. 4, Code 4105. Mechanical Properties Data Center, Belfour, Stulen, Inc. pp. 1-28

Mager, T. R., and R. S. Aspden. Effects of Thermal History and Microstructure on the SCC Behavior of Inconel X-750 in Aqueous Systems at 343°-360°C. Westinghouse Research and Development Center.

Mills, W. J. (1980). Metallurgical Transactions, 11A, 1039-1047.

Mills, W. J. (1981). In L. N. Gilbertson and R. D. Zipp (Eds.), Fractography and Materials Science. ASTM TP 733. pp. 98-114.

Private communication with Huntington Alloys, Inc. (1983). Huntington, West Virginia.

Rao, R. S., and J. M. Alexander (1983). Metals Technology, 10, 14-19.

Raymond, E. L. (1967). Met. Trans. of AIME, 239, 1415-1422.

Server, W. L., D. R. Ireland, and R. A. Wullaert (1974). Dynatup Technical Report No. TR-74-29R. Effects Technology Inc., Santa Barbara, California.

Sinha, A. K., M. G. Hebsur, and J. J. Moore. The effects of aging between 704°C-871°C on microstructural changes in Inconel X-750. Paper presented at the International Symposium on Metallography and Corrosion, Calgary, Alberta, Canada, on July 27, 1983.