The Analysis of Local Plasticity during Macro-indentation of the Nickel-based Alloy 600 Used in CANDU Steam Generator Tubing

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Abstract

Nano-indents were performed on the cross-sectional plane of the macro-indent made in Inconel-600 alloy to investigate the plastic flow behavior of material in plastic zone around it. Average hardness values were calculated in three directions i.e. vertical, horizontal and diagonal. Both, calculated average hardness and von-mises equivalent plastic strain (obtained from FEM modeling) decrease with increasing distance. Yield stress and von-mises equivalent plastic strain in three directions coincide with the flow curve of the material implying that this type of testing technique can be used to extract the material plastic flow curve as obtained from a conventional mechanical testing method.

1. Introduction

The nickel-based alloy Inconel-600 (referred to as Alloy 600 in this work) is designed for use at temperature ranging from cryogenic to 2000°F (1093°C). The high nickel content of this alloy provides a level of resistance to reducing environments, while the chromium content provides resistance to weaker oxidizing environments. The high nickel content of the alloy also provides exceptional resistance to stress corrosion cracking in chloride-rich environments. The chemical composition of Alloy 600 consists of nickel (72%), chromium (14-17%), iron (6-10 %), carbon (0.15 %), manganese (1 %), sulphur (0.015 %), silicon (0.50 %) and copper (0.50 %).

The nickel-based Alloy 600 is used as tubing material in the out-of-core secondary cooling systems found in many types of nuclear reactors. It has excellent resistance to corrosion in high-purity water, and a high resistance to chloride-ion accelerated stress-corrosion cracking in reactor water systems. Alloy 600 steam generator tubes and hardware were used in nuclear reactors for electric power generation beginning in the 1950's and continue to be used in this application. One particular area where the Alloy-600 is used is for tubing within the steam-generator assemblies of CANDU nuclear reactors. These tubes are subjected to high temperature pressurized water and hence could be exposed to conditions where localized regions of the tubing (i.e. regions where stress/strain concentrations exist) where Stress Corrosion Cracking (SCC) could occur. The onset of SCC is known to initiate as a combined result of an applied

driving stress and a favourable microstructure. It is therefore potentially very useful to have a testing technique that can apply a high mechanical stress to a localized region of a component and then expose this stressed region to a corrosive environment to measure its resistance to SCC. Micro-indentation, involving indenting a material to a depth of about 100 μ m, to invoke a small region of high residual stress followed by emersion in a high-temperature caustic solution has been proposed as a way to test local variations in the SCC resistance of the Alloy-600.

In this research, we attempt to come up with a reliable analysis of the extent of local stress resulting from localized mechanical deformation induced by conical indentation in Inconel600 alloy in order to understand the role of this local stress on the initiation of stress corrosion cracking which is a life-limiting factor in steam generator tubing used in the CANDU nuclear reactors. For this purpose, we have employed using nano-indentation hardness testing method to map the local stress distribution in the plastic zone around the macro-indent.

2. Experimental Procedure

Two Inconel Nickel Alloy-600 specimens, 10mm length, 10mm height and 5mm width, were prepared from a bar, originally having 140mm in length and 5mm in width. Prior to polishing, samples were mounted in Bakelite. Polishing was carried out by grinding the samples on different grit papers having grain size of $300\mu m$, $600\mu m$, $1200\mu m$ and $4000\mu m$. A macro-indentation, conical Rockwell C, was made on the polished near to the edge as shown in Figure 1.



Figure 1: Conical indent made with Rockwell hardness testing having approximately 300µm depth.

To investigate material plastic flow of material around this macro-indent, sample was taken out from bakelite mold, to section it through along the cross-section plane of the indent. Excessive

material was removed by polishing procedure mentioned above in the text. Final polishing was carried out up to 0.05µm level using colloidal Alumina. Figure 2 shows the final prepared surface from the cross-sectional plane.



Figure 2: Cross-sectional view of indent after polishing.

Nano-indentation tests were carried out by using Nanotest platform (Micro Materials Limited, Wrexham). An increasing force, F, was applied at a constant-rate, 10mN/sec, to pyramidal Berkovich indenter, until a specified depth of 1 μ m was reached and then the load was held constant for 10 Seconds. Finally, the load was decreased at the original loading rate. System thermal drift was estimated by holding the force constant at 90% of the maximum force value during unloading section of the experiments. The final data were corrected for the estimated instrumental drift.



Figure 3: The nano-indentation matrix around the Rockwell C marco-indent in Nickel-alloy 600.

Since the Rockwell C indentation is symmetrical, the nanoindentation test matrix was constructed on half of the region around it. A matrix consisting of 1496 nanoindents was performed in the selected area. Area was divided in three regions as above shown in Figure 3. Region1 consisted of 36*36 matrix having 1296 nano-indents with 25µm of inter-indent spacing. The data from indent sites marked in rectangular boxes of Region 1 were not included in the final analyses due to incorrect hardness values as a result of equipment error. Region 2 and 3 consisted of 5*20 and 10*10matrix respectively, having 100 nano-indents each with inter-indent spacing of 100µm also shown in Figure 3.

3. Results & Discussion:

Representative indentation force, F, versus distance, h, curves are included in Figure 4.



Figure 4: Load vs. Depth curve

The indentation hardness, H, is measured from obtained force-distance curves by the following expression;

$$H = F/A(h_p), \tag{1}$$

here, $A(h_p)$ is the projected contact area of the indenter, calculated from the plastic indentation depth, h_p , from a F-h curve and for a Berkovich indenter is given by $24.5h_p^2$. Indentation plastic depth, hp, was calculated by making use of Oliver-Pharr model [1].

To analyze the material flow behavior systematically around the macro-indent, the plastic flow region is divided in three directions, namely; Vertical, Diagonal and Horizontal. Three regions are shown in Fig. 5.



Figure 5: Indents marked in rectangular boxes in Horizontal, Vertical and Diagonal directions for calculating average hardness.

The following Figure 6 shows Average Hardness versus distance plot in three chosen directions; (a) Veritcal, (b) Horizontal and (c) Diagonal. It clearly indicates that the average hardness decreases with increasing distance away from the macro-indent boundries. Also, included in the plots are the equivalent von-mises plastic strain values, obtained from FEM analyses [4], which also show the similar decreasing trend as the average hardness values in all the three directions. This is consistent with previous findings by Chaudhri et al. [2-3], who reported that indentation hardness/stress and equivalent plastic strain around spherical and Vickers indentations decreased gradually with increasing distance from the indentation boundaries.



Figure. 6: Average Hardness and Von Mises Eq. Plastic Strain versus distance in; (a) Vertical, (b) Horizontal, (c) Diagonal directions.

The hardness, H, yield stress , σ_{y} , and plastic strain, ϵ , are related by following expression;

$$\sigma_{\rm y} = H/3 = K \epsilon^{\rm n} \tag{2}$$

Where 'K' is a strength coefficient and 'n' is the strain-hardening exponent which varies from 0 to 0.5. The trends in Figure 6 are consistent with this expression i.e. both the hardness and plastic strain show the similar decreasing behavior with increasing distance away from the macro-indent with in the plastic zone around it.

Since the hardness changes with the distance with in plastic zone, Equation 2 indicates that the strain also changes with the distance. In Figure 7, the estimated average hardness values are plotted against the von mises equivalent plastic strain from Equation 2. The curve is similar to the flow-cruve obtained from a uniaxial tensile/compression test. Also, included in the figure is the uniaxial tensile test data on Nickel-600 alloy [5].



Figure 7: Average hardness vs Plastic Strain



Figure 8: Yield Stress vs Strain

In Figure 8 the estimated yield stress values are plotted against the von mises equivalent plastic strain from Equation 2. The curve is similar to the flow-cruve obtained from a uniaxial tensile/compression test. Figures 7 and 8 illustrate that the indentation testing technique can be used to extract the material plastic flow curve as obtained from a conventional mechanical testing method. In advantage, this technique can be used over the specimens at small scale where it is not feasible to extract the standard specimen for tensile/compression testing.

4. Conclusions

This research work presents the study of plastic zone around the macro-indent made in nickel alloy 600. To investigate the plastic flow behaviour of material , nano-indents were made on the cross-sectional plane of the macro-indent and average hardness was calculated in three directions i.e. vertical, horizontal and diagonal. The calculated average hardness and von mises equivalent plastic strain (obtained from FEM modelling) decreases with increasing distance. In Figure 8 yield stress and von-mises equivalent plastic strain in three directions coincides with the flow curve of the material which implies that indentation testing technique can be used to extract the material plastic flow curve as obtained from a conventional mechanical testing method .

References

- [1] W.C. Oliver and G.M. Pharr: "Improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments". J. Mater. Res. 7, 1654, 1992.
- [2] M.M.Chaudhri 'Strain hardening around spherical indentations' *Phys. Stat. sol.* (a) Vol.182, 2000, pp 641-652.
- [3] M.M.Chaudhri 'Subsurface strain distribution around Vickers indentations in annealed polycrystalline copper' *Acta mater.*, Vol. 46, No. 9, 1998, pp 3047-3056.
- [4] A.Z.M. Ariful Islam, Mater of Engineering Science Thesis, The University of Western Ontario, Canada, 2010
- [5] J.F.Hall, J.P.Molkenthin, P.S.Pervey and R.S.Pathania "Measurement of Residual stresses in Alloy 600 pressuerizer penetrations" <u>Conference on the contribution of Materials</u> <u>investigation to the resolution of problems in pressurized water reactors</u>, 12-16 September 1994, Paris.