Metallurgical Defect: 
Manufacturing of a Reference Specimen for NDE Studies

Izella SALETES 1, Tobin FILLETER 2, Anthony N. SINCLAIR 1

1Ultrasound NonDestructive Evaluation Laboratory 
2NanoMechanics and Materials Laboratory

Department of Mechanical and Industrial Engineering, University of Toronto; Toronto, Canada
Phone: +1 416 978 1271; e-mail: saletes@mie.utoronto.ca, filleter@mie.utoronto.ca, sinclair@mie.utoronto.ca

Abstract
A cold shot in a turbine blade could lead to the fracture of the blade as a result of fatigue and then to the failure of the powerplant. Currently, there is no non-destructive method to detect cold shots. Moreover, the development of such a NDE method is hampered by the lack of experimental samples. In the current project, we are developing reference specimens to mimic the ultrasonic response provided by the boundary layer of a cold shot. The specimens are examined to assess their similarity to a cold shot found in a fractured blade. Ultrasound tests are conducted to assess the detectability of the defect.

Keywords: cold shot, ultrasonic NDE, casting defect, turbine blade

1. Introduction

The presence of a casting defect known as a cold shot in a turbine blade that has been installed in a turbine disc in an aircraft engine would weaken the blade, which could then fracture as a result of fatigue. Such occurrences are extremely rare, but a blade fractured in this way would cause engine imbalance, and could lead to the fracture of other blades by impact damage and tensile overload, followed by an In-Flight Shut Down (IFSD) of the power plant.

A cold shot appears during the die casting process, when a small portion of molten metal inadvertently comes into contact with the die and rapidly cools. It has the same chemical composition and a similar microstructure that of the parent bulk material and is typically separated from the bulk alloy material by a thin layer of oxide. Currently, there is no non-destructive method to detect cold shots. Moreover, the development of such a NDE method is hampered by the lack of experimental samples. The only known samples in existence are those from a very few severely distorted and broken blades that have failed in-service.

The examination of those blades showed that the cold shot is typically spherical with a diameter of approximately 1mm, surrounded by an oxide layer of about 1-2μm in thickness. Scanning Electron Microscopy (SEM) investigation shows that there is no delamination between the oxide layer and cold shot, nor between the oxide layer and rest of the blade. There is no evidence of cracking or significant porosity that could aid in nondestructive detection of this type of defect.

We have developed a technique for manufacturing reference specimens with an internal oxide layer which mimics the interfacial layer of a cold shot. The idea was to create an implanted plane oxide layer in a similar nickel-based alloy to that used in turbine blades. Each reference specimen consists of two adjacent, concentric discs. The interface between the discs has two distinct regions: one region with a thin interfacial oxide layer, and one region without oxide. Based on these specimens with manufactured internal "flaws", various NDT techniques can be evaluated for Probability of Detection (POD) of cold shots.

As a first step to finding a suitable NDT technique, high frequency ultrasonic tests are conducted to assess the detectability of the layer. A focused transducer with a nominal central frequency of 100 MHz is used to scan the interface of the specimen. Another transducer with a nominal frequency of 25 MHz is used to measure the amplitude ratio of the first to the
second back-wall echo of the specimen on regions with and without oxide for comparison purposes. A theoretical 1-D model is also used to quantify the reflection coefficient of the oxide layer as a function of frequency, as well as the multiple reflection-transmission combination of the 3-layer system [1, 2, 3].

2. Manufacturing Procedure

A cold shot will normally exist inside a blade with no material delamination that would indicate its presence. The first step of this project was to manufacture a test system with an embedded oxide layer similar to that of a cold shot in terms of thickness and texture, but in a large planar geometry on which various NDE methods can be tested. Figure 1 shows a schematic diagram of the test specimen. Two Inconel-600 discs of different thicknesses (1.5mm and 5mm) are polished on one face with SiC paper (finish polishing with grit #600, #800 or #1200). The oxide is grown on the prepared surface of the thick disc by heating. A literature review and several trials [4] revealed that an oxide layer simulating that found in a cold shot could be achieved by heating a sample disc to 1000°C for 60 minutes, followed by slow cooling. The oxide is then removed on the periphery of the disc by polishing, in order to provide a point of comparison for any NDE tests. The oxidized disc is then electron beam welded to the other polished disc along their peripheries, and subjected to Hot Isostatic Pressing (HIP) at 1120°C, 124 MPa for 4 hours at elevated temperature to bond them together to form one thicker disc. This yields an embedded one-micron layer of oxide that mimics the boundary layer of a cold shot that could be generated during blade casting.

![Figure 1. Schematic diagram and corresponding picture of the specimen. The two discs are then welded and hot isostatic pressed together.](image)

3. Specimen Embedded Oxide / Cold Shot oxide Comparison

Some specimens were cut open in order to examine the cross section with a SEM. A x40 magnified view of the boundary between the regions with and without oxide is presented in Figure 2a. Both regions are shown at magnification x5000 in Figures 2b and 2c. The oxide layer regions (Figure 2b) meet the expectations from the oxidizing study, in that they appear in all respects to closely resemble the oxide layer that surrounds a cold shot, as shown in Figure 3: there is no delamination, no cracks and no porosity, and the thickness is of the order of 1µm. Regarding the region without oxide, the interface is not visible at x40 magnification. However, at x5000 magnifications, Figure 2c shows that the interfacial region where there is no prepared oxide layer is not actually constant in thickness; Energy Dispersive X-ray measurements confirm that the dark areas are not oxide but relief due to the surface roughness of the discs before welding and the HIPping process.
4. Ultrasound Measurements

The first NDE methods tested were ultrasonic inspection of the oxide at high frequencies. Experiments were carried out in a water tank in which a focused piezoelectric transducer was immersed and placed normal to the surface of the specimen.

4.1 Reflection of the interface with 100MHz transducer

An immersion probe with nominal central frequency of 100MHz (diameter: 5.6mm, focal length: 12.7mm) was positioned to have the focus point at the interface between the 2 original discs. At such a high frequency, the attenuation is too high to see an echo from the back-wall. According to the velocity in the alloy, the interface echo should be 1.096µs after the front-wall echo. Neither the raw A-scan, nor the FFT spectrum shows any discernible echo from the oxide at the interface.

4.2 Amplitude ratio of the 1st to 2nd back-wall echo with 25MHz transducer

The second probe was 6.4mm in diameter, 73mm in focal length, with a nominal central frequency of 25MHz. The focal point was placed 2mm after the back-wall in order to have maximum energy on the 2 first back-wall echoes. Figure 3 shows the amplitude ratio of the first back-wall echo to the second back-wall echo. The area with oxide is distinguishable from the area without oxide. However, the contrast between both regions is slight. The echo indications from the periphery of the specimen are due to the welding and surface roughness on the outer surface.
5. Theoretical 1-D propagation

A 1-D model of the propagation of an acoustic wave through a 3 layer system, where the center layer is very thin, is proposed. The model is based on ray tracing between the interfaces, and the net effect on the reflection and transmission factors [1, 2, 3] and will be used for 2 purposes. First, the reflection coefficient of a thin oxide layer can be plotted as a function of the frequency, which will provide the optimum frequency for the detection of the oxide layer. Second, the reflection and transmission factors of the entire system will give the effect of the oxide layer interfaces on the amplitude of the back-wall echoes. This model depends on the acoustic impedance difference between the nickel-alloy and the oxide. Mechanical properties of the nickel-alloy are well known and are easy to measure. However, density and sound velocity of the oxide for Inconel-600 have not been reported in the literature. Therefore, experiments were carried out in order to determine the density and the Young’s modulus of the oxide.

5.3 Density measurement of the oxide

Density of the Inconel-600 oxide was deduced from SEM measurements. The EDS gave the approximate number density of each elemental component, from which the density was calculated. The density of the bulk alloy without oxide was also measured in order to validate the method. The theoretical density of the nickel-alloy is \( \sim 8500 \text{kg/m}^3 \) whereas the EDS measurement yielded 8535kg/m\(^3\). The density of the oxide was evaluated from 4 measurements which gave a mean value of 4130kg/m\(^3\) and a standard deviation equal to 100kg/m\(^3\).
5.4 Young’s modulus measurement of the oxide

Stiffness measurement of the oxide is hampered by the very small thickness of the layer. One method being pursued for conducting this measurement is nanoindentation [5]. Some preliminary tests were performed which indicated that the stiffness of the oxide is very high; the nano-indentation experiments must therefore be repeated with a diamond tip in order to get meaningful results.

We are also attempting to estimate the modulus with an atomic force microscope (AFM), using a silicon tip. Again, the high modulus of the oxide rendered these preliminary measurements unsuccessful. Additional measurements are planned using a harder tip for the AFM.

6. Conclusion

A manufacturing procedure was developed to mimic the interfacial oxide layer which characterizes a cold shot. The examination of the cross section of both specimen and cold shot with a SEM validates the manufacturing method. Ultrasound tests were carried out to try to see the manufactured defect. Very high frequency (100MHz) ultrasound was not able to detect the 1µm oxide layer, whereas 25MHz attenuation measurements are encouraging. Young’s modulus of the oxide has to be determined to allow proper development of a 1-D model of wave propagation in the specimen. This is being attempted by AFM and nanoindentation with the use of a diamond tip.

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References