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Revision 0

Status Report: Design and Fabricate Installation-ready Test Capsule for the In-situ Measurement of Elastic Properties Using an Optical Fiber-based Measurement Technique

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March 2019

Idaho National Laboratory

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EXECUTIVE SUMMARY

Microstructure evolution due to irradiation in a nuclear reactor can have a dramatic effect on material properties. A better understanding of this evolution is necessary for developing improved nuclear fuels and materials. The ability to measure such changes in real time is extremely limited due to the harsh conditions, high radiation fields, and limited access of the reactor environment. Through carefully designed experiments, measurement of elastic properties can be tied directly to microstructure. We present the methodology, design, and laboratory test results for an instrument that has been developed to monitor grain microstructural changes during irradiation. Our measurement approach involves exciting and measuring the resonant frequency of a thin cantilever beam. Excitation and detection of the flexural vibrations of the beam are accomplished using optical methods that require only an optical fiber connection between the instrumentation and the sample. This technique has been demonstrated in a laboratory setting to monitor the recrystallization of highly textured copper during high temperature annealing. A test capsule incorporating this technique has been developed for in-reactor testing. The capsule has been designed to be compatible a reusable test module that allows simplified insertion in the TREAT reactor at INL. An exact replica of the instrument described in this Status Report, constructed from Quality Level 3 materials, is slated to be inserted into the TREAT reactor in FY19.

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1. INTRODUCTION

In-pile monitoring of microstructure evolution of nuclear fuel would provide the computational materials science community direct access to a time-dependent, in-reactor environment. It would also enable monitoring in-pile material behavior that cannot be captured in a post-irradiation environment. This has motivated development of facilities that combine ion beam and electron beam irradiation with transmission electron microscopy. However, creating neutron beams that have a flux and energy distribution representative of in-pile conditions is currently not possible, and, thus, a comparable capability to examine the influence of neutron irradiation on microstructure evolution does not currently exist. To close this capability gap will require development of innovative instruments that can indirectly measure changes in microstructure. The work presented here describes the development of an in-pile laser resonant ultrasound spectroscopy method to measure changes in elastic properties caused by changes in microstructure. Ultimately, this approach will require preforming the inverse problem of relating elastic properties to the state of the microstructure. The ability to monitor a recrystallization event is demonstrated.

Microstructure evolution in nuclear fuel is governed by atomic transport facilitated by a highly non-equilibrium distribution of Frenkel defects (vacancy – interstitial pair). Over time in reactor, vacancies coalesce, forming small voids. Eventually, these voids are filled with fission gas. Transport of fission gas bubbles through the fuel plays a central role in eventual fuel failure. The interstitial portion of the Frenkel defect is preferentially drawn to dislocations, causing dislocations to multiply. The strain energy associated with dislocation production coupled with high temperatures causes profound changes to the grain microstructure. Grain restructuring or recrystallization occurs in both oxide and metallic fuel and can cause dramatic changes in material properties [1,2,3]. It has been shown previously that the elastic properties of materials are strongly influenced by porosity and grain microstructure. Here we are interested in measuring changes in elastic properties brought about by changes in grain texture.

The real time measurement of microstructure-mediated elastic properties in a reactor is a challenging task. The environmental conditions in the reactor include very high radiation, with high neutron fluences in both the fast and thermal regimes, as well as high gamma radiation, in addition to high temperature and pressure. Material testing positions in a reactor also tend to be small with little space for instrumentation and limited access. Long cable runs are usually required and often must pass through bulkhead fittings at pressure boundaries. Material testing is commonly done using large instruments or fixtures unsuitable for in-reactor testing and most sensors are not able to withstand in-reactor radiation levels. Indeed, the majority of microstructure characterization measurements are currently made using post-irradiation examination (PIE) techniques which provide only before/after information and lack the ability to measure changes in materials as they occur.

In this work, we present the measurement methodology, design, and laboratory testing of an instrument that will be used to monitor grain microstructural changes of a sample during irradiation in a nuclear reactor. Our approach will involve relating changes in polycrystalline elastic properties to grain microstructure. First, a highly textured grain microstructure will be introduced into a copper sample by rolling. Then, the grain microstructure will be monitored through the microstructure-induced elastic property changes of the sample as it undergoes recrystallization. The measurement technique involves exciting and measuring flexural vibrations in a thin cantilever beam using optical fiber transmission for excitation and detection. The device has been designed to be compatible with the Minimal Activation Retrievable Capsule Holder (MARCH) that will allow simplified testing in the Transient Reactor Test (TREAT) Facility at Idaho National Laboratory (INL). The planned test will assess the impact of one or more high neutron flux transients on the recrystallization temperature of the copper sample.

2. MEASUREMENT APPROACH

2.1 Resonant Beam Vibrations

The approach for monitoring microstructure changes in-pile consists of repeatedly measuring the resonant frequency of a vibrating beam fabricated from the material of interest. Although this technique has been demonstrated in laboratory experiments conducted previously [4], the basic concept is presented here for completeness.

The natural frequency of vibrating beams has been widely used as a means of determining the elastic properties of materials, and numerous standards exist detailing such measurements [5,6,7]. For an isotropic material, the natural frequency depends only on the beam geometry, density, and elastic properties. Free-free beams are generally specified in these standards; however, cantilever beams offer advantages for in-reactor measurements because the beams can be held rigidly in position and alignment with the detection system can be maintained.

Calculation of the natural frequency for flexural vibrations of a cantilever beam has been broadly discussed in the literature [8,9,10]. The simplest formulation is based on Bernoulli-Euler analysis which neglects shear deformation and rotational inertia. Calculation of the natural frequencies based on Bernoulli-Euler formulation can be performed using *Equation (1)* below [11]:

$$f_n = \frac{(\beta_n l)^2}{2\pi l^2}\sqrt{\frac{EI}{\rho A}}; \quad \text{with} \quad (\beta_1 l = 1.875,\ \beta_2 l = 4.694, \beta_3 l = 7.855, \dots) \tag{1}$$

where l is the beam length, E is the modulus of elasticity, I is the geometrical moment of inertia about the bending axis, ρ is the density, A is the cross sectional area, and $\beta_n l$ are solutions to the frequency equation for successive flexural modes with n being the mode number. While less accurate than other theories such as Timoshenko beam theory, this simple formulation gives accuracy to within a couple percent when $(n/l) * (I/A)^{1/2} < 0.01$, which is sufficient for monitoring the elastic property changes described here [12].

2.2 Optical Excitation and Detection

Optical methods are used for excitation and detection of the beam vibrations which allows for transmission to the sample via optical fibers. Figure 1 is a cross section view of the test capsule showing the relationship of the cantilever beam and the fiber optic excitation and detection probes. Excitation is accomplished using an amplitude modulated laser. The laser light is coupled into an optical fiber and delivered to the base of the beam as shown in Figure 1. Excitation occurs through optical heating and the resulting thermal expansion. A network analyzer controls the modulation frequency that is swept over a range including the resonant mode of interest. Detection of the beam deflection is based on a fiber optic lever technique [13]. Detection light is provided by a broad band source coupled into an optical fiber. It passes through a bi-directional fiber splitter and is then delivered to the tip of the vibrating beam (see Figure 1). Light exiting the optical fiber probe is reflected from the sample surface and coupled back into the fiber where it propagates back toward the source. The light exits the fiber tip in a divergent cone determined by the numerical aperture of the optical fiber. The intensity of the light returning to the core of the optical fiber is dependent on the distance between the fiber tip and the sample. Thus, deflection of the cantilever beam causes an intensity modulation of the light propagating back toward the source. On its return, the intensity modulated light encounters the beam splitter where a portion is diverted to a photoreceiver. The photoreceiver signal is processed and recorded as a function of modulation frequency by the network analyzer. Since we are primarily interested in the frequency of vibration, the absolute intensity of the light is unimportant as is an absolute calibration of the deflection. This detection technique requires only a broadband light source, a fiber splitter, and a photoreceiver. Its sensitivity is in the sub-micrometer range which is not as sensitive as an interferometer, but is less complex, and requires no stabilization or expensive optics. Since this detection method does not rely on the absolute light intensity, the measurement is only minimally affected by radiation-induced attenuation in the optical fiber – making it a good candidate for in-reactor measurements.

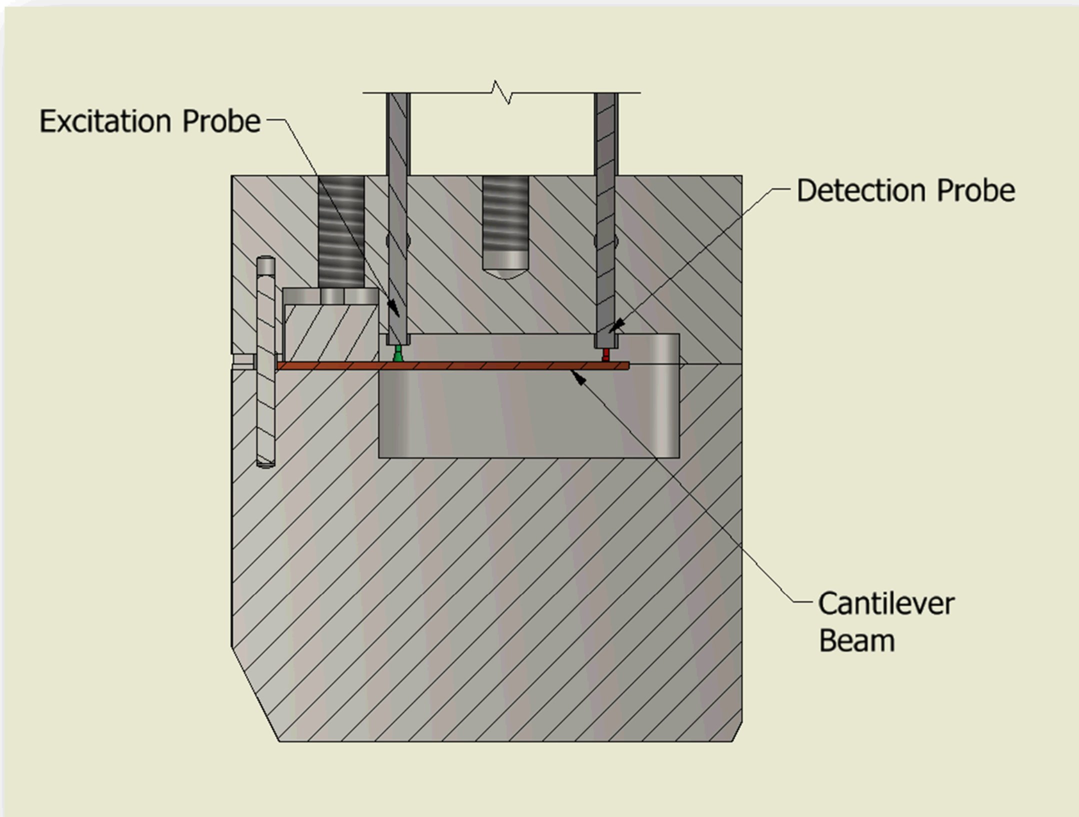

Figure 1. Section view of the test capsule showing the cantilever beam with excitation and detection probes.

3. DEVELOPMENT OF INSTRUMENT CAPSULE

Development of an instrument for actual in-pile measurements required the design of an experiment capsule compatible with the test position in the TREAT reactor. The primary functions of the capsule include first, the ability to precisely clamp the cantilever beam in position, and second, to hold the optical fiber probes which deliver the light to excite and detect the beam vibrations. A cross section view of the capsule design was shown in Figure 1, and Figure 2 shows a cut-a-way view of the capsule assembly along with photos of the capsule. The capsule was fabricated from a 1.45 in. diameter titanium bar and consists of upper and lower halves. Titanium was used to minimize radioactive activation. The top surface of the lower half of the capsule includes a central pocket with a shallow slot at one end. The slot is used to align the beam, and the pocket allows the space for the beam to vibrate. A pin located near the back of the slot serves as a reference for beam indexing in the slot. Pins on either side of the slot guide a small block that is used to clamp the beam. The upper half of the capsule is bolted to the lower half using four socket head cap screws. A threaded hole in the upper half supports a set screw that is used to supply the clamping pressure to the beam. Two 1/16 in. diameter holes in the upper half are used to position the optical fiber probes over the base and tip of the cantilever beam. The probes are held in position using set screws that thread into the side of the capsule. An additional hole allows a thermocouple to be positioned in the capsule, adjacent to the beam. A threaded hole in the center of the upper half is used to attach the capsule to the support rod.

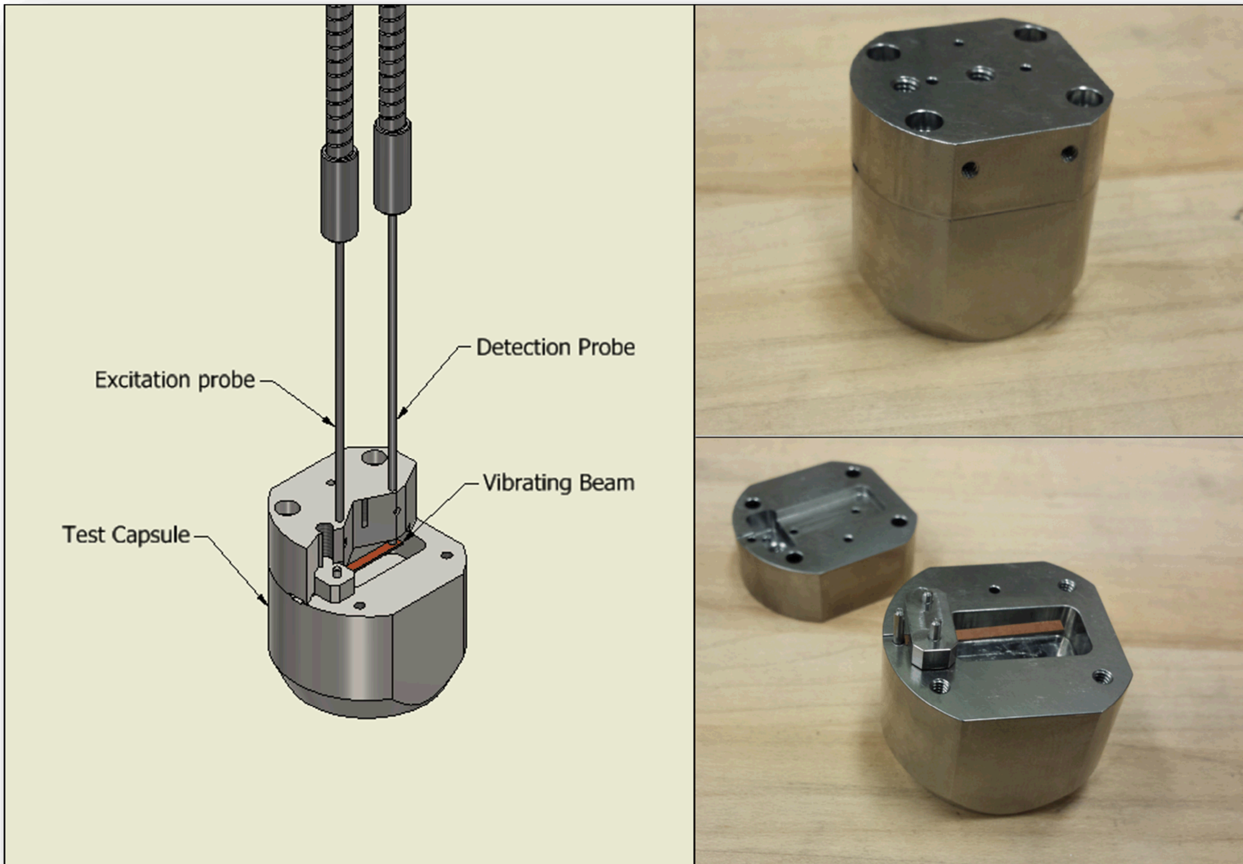

Figure 2. Cut-a-way view of the test capsule (left) and photographs of the fabricated test capsule (right).

3.1 Laboratory Test Results

The instrument capsule described in the previous section has been fabricated, assembled, and tested in the laboratory to verify functionality and suitability for in-reactor testing. In this laboratory experiment, a highly textured copper sample was incorporated into the instrument capsule and heated on a hot plate, while the resonant frequency was monitored using the technique described above. The resonant frequency vs. temperature as the experiment was heated above the recrystallization temperature and subsequently cooled are shown in Figure 3. Note the sharp drop in the resonant frequency as recrystallization began at about 177°C. A plot of the signal amplitude vs. frequency for the first flexural mode before and after heating are shown in Figure 4. A significant decrease in the resonant frequency occurred at the transition temperature, indicating a microstructural change that affected the elastic properties of the sample. This annealing or recrystallization resulted in a shift of the elastic modulus from about 118 GPa to 71 GPa.

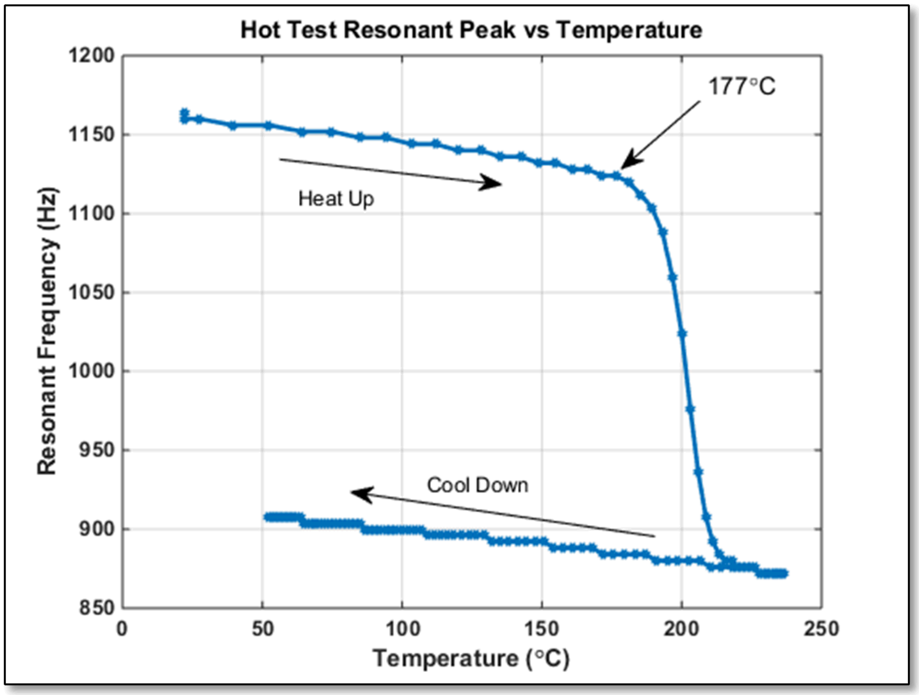

Figure 3. Resonant frequency vs temperature.

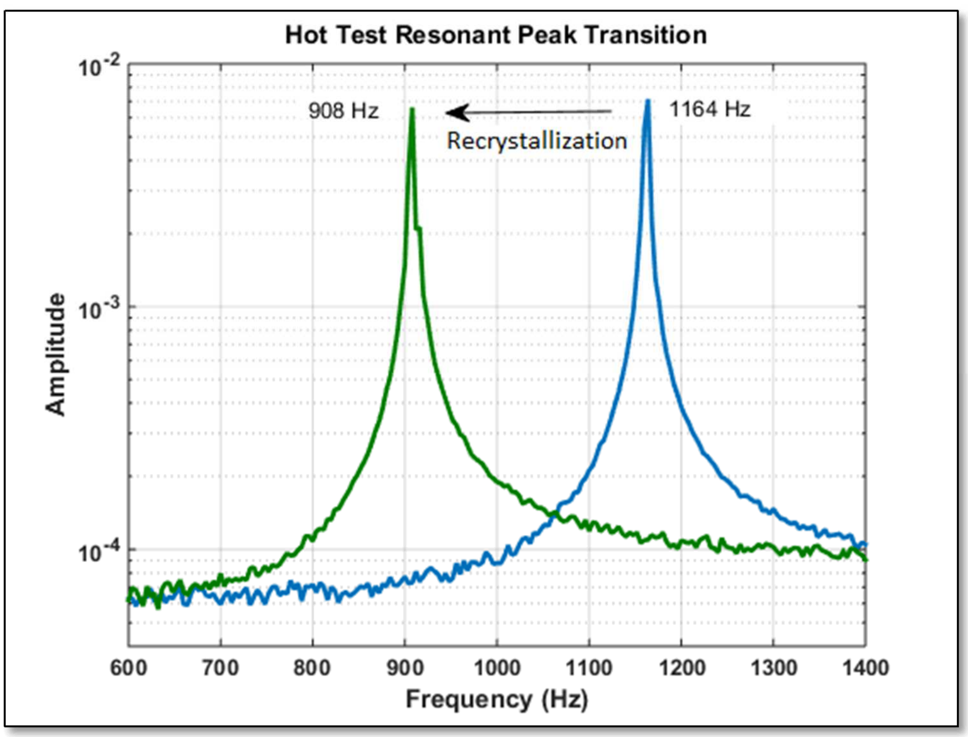

Figure 4. Resonant frequency shift resulting from recrystallization.

4. SUMMARY

Grain restructuring or recrystallization occurs in both metal and ceramic nuclear fuels during irradiation and can result in dramatic changes in properties. Measurement of elastic properties can be tied directly to these changes in microstructure. In this paper, we have described the methodology, design, and laboratory testing of an instrument that will be used to monitor microstructural changes of a copper sample with a highly textured grain microstructure during irradiation. The approach involves monitoring the resonant frequency of a thin cantilever beam using optical methods for excitation and detection. An exact replica of this capsule, constructed from Quality Level 3 materials, is slated to be inserted into the TREAT reactor in FY19.

5. REFERENCES

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