

Article

Acoustic Nonlinearity and Loss Associated with Unbonded Interfaces and Dislocations in Additively Manufactured CoCrMo Parts with Complex Geometries [†]

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Abstract

Complex geometries of additively manufactured (AM) metal parts present challenges for rapid post-build part qualification with most conventional nondestructive measurement techniques. This study explores the potential of nonlinear reverberation spectroscopy (NRS) for detecting defects in parts with complex geometries. It focuses on measurements of two CoCrMo hollow pentagonal star-shaped specimens with and without designed-in mesoscale cavities and trapped unmelted powder. Nonlinearity and loss of the specimen with cavities are found to be greater than those of the specimen without cavities, and these differences are found to increase after six years of aging at room temperature. Different behavior of the two specimens with respect to aging and extended acoustic excitation lead to the conclusion that the dominant sources of nonlinearity and loss in the two specimens are different: contacting unbonded internal interfaces in the specimen with cavities, and dislocations in the other specimen. This conclusion is partly supported by calculations showing that the time scale of an aging-induced decrease in nonlinearity of the specimen without cavities is consistent with expected rates of migration of vacancies and nitrogen interstitials to dislocations. The results support the idea that NRS measurements would be useful for rapid nondestructive qualification of AM parts with complex geometries.

Keywords: additively manufactured metals; nonlinear reverberation spectroscopy; nondestructive evaluation; nonlinearity; acoustic loss; CoCrMo; complex geometry; vacancies



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1. Introduction

Additive manufacturing (AM) of metal parts offers numerous technical and financial advantages relative to traditional production methods, including direct generation of parts with complex geometries [1]. However, complex geometries present great challenges for nondestructive evaluation (NDE) with most conventional techniques, and post-build NDE is currently necessary to ensure quality of AM parts in applications with critical mechanical requirements [2,3]. X-ray computed tomography (Xray-CT) is generally considered the “gold standard” for characterizing voids in AM metals but is time-consuming, expensive, and limited to parts with dimensions on the order of a couple of centimeters (dependent

on alloy density) if image resolution on the order of ten microns (μ Xray-CT) is to be achieved [2]. Resolutions in the micron range can also fail to provide information on defects that have a critical effect on fracture and fatigue performance of AM parts [4]. The layered build of AM alloys leads to potential presence of lack-of-fusion (LOF) defects with limited height along the build direction and correspondingly low volume. LOF defects with gaps less than a few microns will go undetected in μ Xray-CT with typical resolutions, and such defects are expected to be particularly vulnerable to growth during fracture and fatigue [2,4,5]. While this resolution limitation can be addressed with “nanofocus” Xray-CT, the maximum part dimensions that can be scanned with such systems are only a few millimeters [2].

More rapid and economical NDE techniques for probing internal voids and crack-like flaws (including LOF defects) with mesoscopic or macroscopic cross sections include propagating-wave conventional ultrasonic tomography (CUT) and phased-array ultrasonic tomography (PAUT) [6]; however, these techniques face major challenges with complex geometries. CUT can only detect a defect if: (1) it is in a location where there is a direct interior line of site (LOS) from a point of acoustic excitation to the defect (i.e., no external surface along the line between the excitation point and the defect), and (2) ultrasonic propagation along such a path can be achieved under the constraint of CUT having a fixed angle of the ultrasonic beam relative to the surface normal. PAUT partly addresses the second of these requirements through synthetic beam steering of the ultrasonic wave, but the challenge of detecting defects that do not lie along a LOS path from the excitation remains. This challenge may eventually be partly addressed by including a multi-view total focusing method (MVTfM) with PAUT, but this approach has not, to our knowledge, been pursued with AM metals, and its effectiveness at detecting reflections from defects in some locations is expected to be limited by obscuration of defect signals by surface reflections from multiple wave paths [7].

Experimental demonstration of capabilities and limitations of CUT and PAUT for detecting defects in complex geometries are provided by results from research labs that participated in an ASTM/ISO study of NDE techniques for AM metal parts with hollow pentagonal star-shaped external geometries and designed-in cylindrical and spherical voids with mesoscopic diameters, some containing trapped unmelted powder [6,8–10]. CUT measurements of an aluminum star artifact produced in the ASTM study found the detectability of cylindrical defects with diameters in the range of 0.1 mm to 0.7 mm to be highly dependent on the relative orientations of the cylinders and the excitation surface [9], and some cylinders with diameters less than 0.6 mm were not detected with excitation at any surface. Spherical defects with diameters of 0.1 mm to 0.7 mm containing unmelted powder were also not detected with CUT excitation at any surface. PAUT measurements of this star were found to have greater detection capability, extending the size limit down to 0.1 mm diameter for some cylinder orientations and the spheres, but some cylindrical cavities remained undetected with excitation on any surface.

The intrinsic limitations of propagating-wave CUT and PAUT in complex geometries have led to an exploration of alternate resonant acoustic techniques for NDE of AM parts. Linear resonance techniques explored in the ASTM multi-lab study [10] include PCRT (process compensated resonance testing) and RAM (resonance acoustic method). Rather than attempting to provide information on the presence of specific defects, the tests with these techniques focused on determining whether significant differences in vibrational spectra could be identified in 88 “half-size” stars, including 48 stars with built-in defects and 40 stars with no intentional defects. PCRT measurements successfully sorted all of the stars into pass/fail categories based on acceptable resonant spectra determined from the scatter in resonant frequencies of the stars without defects, and RAM measurements successfully

sorted all but one of the stars into pass/fail categories [9]. Therefore, the complex external and internal geometries of the specimens were not found to be a significant impediment to PCRT or RAM determining the overall presence or absence of defects in these specimens. Note that a pass/fail criterion fulfills the basic objective of rapid quality assessment of AM parts in industrial settings, and acoustic resonance measurements that detect the presence of defects could be followed by more time-intensive destructive or nondestructive tests (e.g., electron microscopy or Xray-CT) to provide spatial information on defects for optimization of AM build parameters.

PCRT, RAM, and other linear resonance techniques rely on the dependence of resonant frequencies on macroscale density and stiffness to detect the presence of voids and/or crack-like flaws. Their accuracy and sensitivity are limited by resonant frequencies also being highly sensitive to variations in external geometry (which is typically less repeatable in AM parts than in machined parts), slight differences in the temperatures of reference and test parts, and potential low volume of performance-limiting defects. Measurements at a single resonance frequency are also insensitive to defects located at anti-nodes of the vibrational displacement pattern, but enhancements in detectability and some information on the locations of flaws can be provided by measurements of several resonant modes [11,12].

Resonance techniques that extract information on acoustic nonlinearity and/or loss offer advantages over linear resonance techniques, as nonlinearity and loss are insensitive to minor variations in external geometry and typically more sensitive to the presence of performance-limiting defects with one or more dimensions below the mesoscopic range, including crack-like flaws and dislocations [13–18]. Therefore, such techniques have been proposed as a basis for rapid cost-effective qualification of AM parts [19–25]. The most widely pursued technique in this category is nonlinear resonance ultrasound spectroscopy (NRUS), which quantifies nonlinearity in terms of fractional shifts in resonant frequency as a function of vibrational amplitude induced at a series of excitation levels [26]. Another technique is nonlinear reverberation spectroscopy (NRS), which extracts information on amplitude dependence of resonant frequency during a single ringdown after tone-burst or impulse excitation [26,27]. Because of the rapid acquisition of data in NRS, uncertainties arising from temperature drift are much smaller than in NRUS, which leads to greater precision of measurements of fractional shifts in resonant frequency than those achieved in NRUS [27].

In our lab at NIST, an NRS technique with electromagnetic–acoustic transduction and phase-sensitive reception has been employed to provide information on resonant acoustic nonlinearity and loss in AM metals [23–25]. An initial NRS study of AM 17-4 stainless steel found a correlation of nonlinearity and loss with porosity that was intentionally introduced through non-optimal laser hatch spacing and quantified with μ Xray-CT [23]. This study also found a correlation of nonlinearity and loss with unmelted powder fraction. Open pores are not expected to directly introduce significant nonlinearity or loss of resonant modes [23,24,28], but hysteretic contact of internal surfaces, including clapping under normal stress and friction under shear stress in crack-like flaws, has been proposed in many studies as a source of nonlinearity and loss [29].

A subsequent more extensive pair of research studies at NIST focused on commercially pure AM aluminum cylinders with volume fractions of porosity in an industrially relevant range of 0.1% to 0.4% [24,25]. Variations in porosity between specimens were introduced by intentional differences in laser power. In addition to NRS and Xray-CT, these studies employed scanning electron microscopy (SEM), Archimedes density measurements, and Ritz calculations of vibrational displacement patterns of two resonant modes that would be degenerate in the absence of slight elastic anisotropy. The acoustic and Archimedes measurements found nonlinearity and loss of these resonant modes to be approximately linearly

correlated with porosity and to be greater for the mode with greater shear gradients across the build plane [25]. These results on anisotropy of nonlinearity and loss, combined with SEM and Xray-CT results, led to a hypothesis that hysteretic movement of surfaces within lack-of-fusion (LOF) defects plays a major role in the observed correlation of nonlinearity and loss with porosity. Analysis of the magnitude of a component of nonlinearity and loss that is independent of porosity led to a conclusion that dislocation nonlinearity/anelasticity is also significant in this material. An additional feature of the results is that the nonlinearity and loss were found to decrease substantially with accumulated duration of acoustic excitation over periods of hours and to pause in these changes when acoustic excitation was paused. This result led to the surprising conclusion that resonant ultrasonic excitation at typical levels employed in nondestructive evaluation of materials induces changes in defects in these AM specimens. This effect is not anticipated to be an impediment to the application of NRS for AM part qualification in industrial settings because only a few minutes are required for measurements.

In this report, we present noncontacting phase-sensitive NRS measurements that demonstrate the capabilities of this technique for characterizing nonlinearity and loss in AM parts with complex geometry. Results are presented for two AM CoCrMo pentagonal star-shaped specimens with and without intentional built-in voids and unconsolidated powder. Measurements performed one year and six years after manufacture of the specimens reveal substantially greater acoustic nonlinearity and loss of the specimen with built-in defects and an evolution of acoustic characteristics that is most pronounced in the specimen without intentional flaws. Hypotheses for the dominant physical mechanisms responsible for the differences and aging of nonlinearity and loss of the two specimens are explored.

2. Specimens

The two AM CoCrMo test artifacts in this study were designed and additively manufactured as part of the large research study organized by ASTM International and the International Organization for Standardization (ISO) that is mentioned in Section 1 [10], which focused on assessing the capabilities of NDE techniques for detecting and locating manufacturing flaws in AM metal parts. The public, private, and commercial labs that participated in this study included NIST and Laboratoire National de Métrologie et d'Essais (LNE). The hollow pentagonal “star” artifacts designed and fabricated in the study included eight alloys and two sizes. Cavities were intentionally built into some of the stars (i.e., included in the CAD model), and some of these cavities contained deliberately unconsolidated powder. The NDE techniques that were tested to determine their ability to detect and locate cavities included Xray-CT, neutron imaging, neutron diffraction, synchrotron radiation CT, thermography testing, ultrasonic testing (UT), phase-array UT (PAUT), process-compensated resonance testing (PCRT), a linear acoustic resonance with impact excitation (RAM), and nonlinear acoustic resonance with impact excitation (NLR). However, only Xray-CT, synchrotron radiation CT, and RAM were employed to characterize CoCrMo stars, only RAM was employed to characterize the two “half-size” CoCrMo stars that are the focus of the current study, and no other half-size CoCrMo stars were built (see Annex D in the ASTM Technical Report [10]). The unpublished RAM results on full-size and half-size CoCrMo stars found the presence of built-in cavities to be reflected in a trend of downward shifts in frequencies of selected resonant modes in a range of tens of kilohertz [30].

The two half-size CoCrMo star artifacts in this study were built at NIST in January 2018 in an M290 EOS laser powder-bed fusion (PBF-LB) system. Note that commercial equipment and materials are identified in this paper for technical completeness; such identification is not intended to imply a recommendation or endorsement by NIST or to

imply that these products are necessarily the best available for the purpose for which they are employed here. The powder composition was 60% to 65% Co, 26% to 30% Cr, and 5% to 7% Mo by weight, matching the composition specified in the ASTM F3213 standard for AM CoCrMo [31] and the ASTM F75 standard for cast CoCrMo [32]. The build parameters included 290 W laser power, 950 mm/s laser speed, 0.11 mm hatch spacing, and 40 μm layer thickness. One star, labeled NIST5, was built without intentional cavities according to CAD design “S0” for half-size stars described in the ASTM Technical Report [10]. The CAD design for the other star, labeled NIST6, has the general configuration of cavities specified in ASTM design “S2” for full-size CoCrMo stars see ([9], Figures 34–38; [10], Figures 23–24), but all external dimensions, cavity dimensions and spacings are reduced by a factor of two relative to that design (see [10], Table D.1).

Figure 1a shows a photograph of specimen NIST6, and Figure 1b shows transverse dimensions of the stars and the regions of the design for NIST6 containing intentional cavities. The build direction is the five-fold axis. The measured length of each specimen along the five-fold axis (not including raised labels) is (22.1 ± 0.1) mm, and the smallest transverse distance between the inner and outer surfaces is (2.43 ± 0.02) mm.

Regions R1–R5 in the design for NIST6 indicated in Figure 1b contain a variety of cylindrical and spherical cavities distributed along the length, with diameters in the range of 100 μm to 400 μm , cylinder lengths in the range of 1.0 mm to 2.5 mm, and various cylinder orientations. (see [9], Figures 34–38, and [10], Figures 23–24, for depictions of full-size S2 CoCr stars). These cavities are intended to be representative of a variety of flaws that can be present in AM parts. Layer defects are represented by cylindrical cavities with the cylindrical axis in the build plane in regions R1 and R2; cross-layer defects are represented by cylindrical cavities aligned with the build direction in R4; and defects containing unconsolidated/trapped powder are represented by spherical cavities containing powder in region R3 and by various orientations of cylindrical cavities containing powder in region R5.

Xray-CT imaging of NIST6 to directly confirm the presence of cavities specified in the CAD design is not pursued in this report. We refer instead to synchrotron-based Xray-CT imaging previously reported for a full-size S2 CoCrMo star from the ASTM-coordinated study with the same starting powder and build parameters (laser power, speed, hatch spacing, and layer thickness) as the specimens in this study. Those results showed that all designed-in cavities with and without entrapped powder were successfully created [9,10]. Therefore, similar effectiveness of the build is expected for NIST6, at least over the range of diameters (greater than 150 μm) of cavities imaged in the full-size stars, which includes all but the smallest cavity diameters (100 μm) in the CAD design of NIST6.

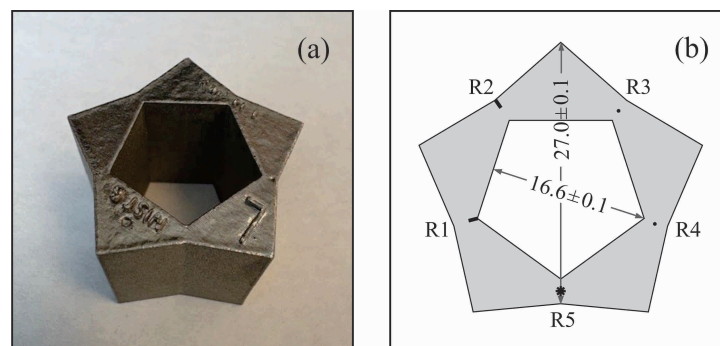


Figure 1. (a) Photograph of “half-size” CoCrMo star artifact NIST6 and (b) diagram of this star showing cross-sectional dimensions in millimeters and regions with built-in cavities along the length. The external dimensions and dimensions of the internal built-in cavities are approximately half of the values specified for full-size ASTM star artifacts with design S2 [9,10].

3. Experimental Technique

3.1. Transduction

The NRS measurements employ electromagnetic-acoustic resonance (EMAR) with tone-burst excitation. A radio-frequency (RF) tone burst with a duration of 4.6 ms is supplied by a RITEC RAM-5000 SNAP system. The burst is passed through an active diplexer to a cylindrical coil containing the specimen in a uniform magnetic field $\mathbf{B}$ with a magnitude of (0.540 ± 0.005) T [27]. The coil consists of 104 turns and has an inner diameter of (41.7 ± 0.3) mm and length of (28.3 ± 0.1) mm. As depicted in Figure 2, the five-fold axis of the specimen, coil axis, and direction of $\mathbf{B}$ are all perpendicular to one another. The orientation of the specimen during measurements has the coil axis vertical and the five-fold star axis horizontal. The specific azimuthal orientation employed in this report is shown in Figure 1b, with the point of the star between R2 and R3 pointing upwards and the axial edges adjacent to region R5 resting on a horizontal surface covered with paper. Paper was selected for the supporting surface because it has a very low acoustic impedance relative to CoCrMo, and as such is expected to transmit very little acoustic energy out of the specimen (i.e., introduce little perturbation to a free boundary condition). Quantitative estimation of the effect of this contact on the acoustic nonlinearity and loss is not pursued in this report, since the two specimens had the same form of support and the focus of the study is on relative differences in the measured nonlinearity and loss of the specimens.

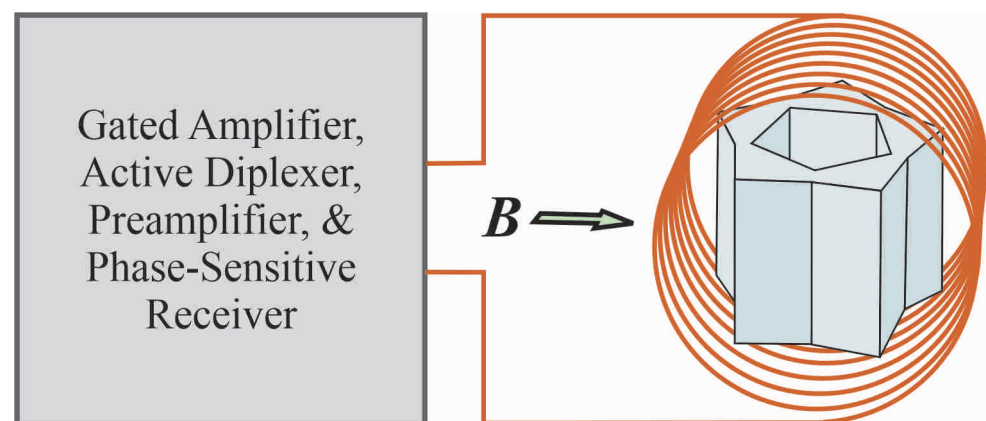


Figure 2. Configuration of the specimen, surrounding coil, and static magnetic field $\mathbf{B}$. The five-fold axis of the specimen, cylindrical axis of the coil, and direction of $\mathbf{B}$ are mutually perpendicular. The number of turns of the coil depicted in this figure is less than the actual number of turns in the coil (104).

With the configuration depicted in Figure 2, near-surface eddy currents induced in the specimen by the RF current in the coil generate Lorentz forces that are primarily aligned with the coil axis and opposite in sign on the left and right sides of the figure. Relatively little force is generated on the planar end surfaces of the specimen because the cross product of $\mathbf{B}$ and induced current in the specimen is close to zero on these surfaces. A similar configuration was previously employed to excite higher-order torsional modes in cylinders of AM 17-4 stainless steel [23].

If the gated sinusoid of the tone burst is chosen to have a frequency f_{ref} close to a resonant frequency of an acoustic mode of the specimen (i.e., if the bandwidth of the tone burst includes the frequency of a resonant mode), then this mode will be excited. Specimen vibration after tone-burst excitation is then detected through the inverse Lorentz effect, with voltages that are induced across the coil passed through the diplexer and a preamplifier to the input of the RITEC receiver [27].

3.2. Signal Analysis

The RF signal received from the coil is passed within the RITEC system to electronic components that act in a manner similar to a vector voltmeter to extract the in-phase and out-of-phase components of the detected signal as a function of time t during resonant ringdown, $PhDet1(t)$ and $PhDet2(t)$, respectively, relative to the sine wave that was gated to produce the tone burst [27]. These components vary much more slowly than the RF signal. They are passed through a 50 kHz low-pass filter to reduce noise, then digitized and passed to a computer.

$PhDet1(t)$ and $PhDet2(t)$ are corrected for slight receiver nonlinearity, time-dependent backgrounds, and gain imbalances that are measured in the absence of a specimen [27]. Instantaneous RF amplitude $A_{RF}(t)$ across the coil and phase $\phi(t)$ are then calculated from these components [27]:

$$A_{RF}(t) = 10^{-G_{dB}/20} \left(PhDet1(t)^2 + PhDet2(t)^2 \right)^{1/2}, \quad (1)$$

$$\phi(t) = \tan^{-1} \left(\frac{PhDet2(t)}{PhDet1(t)} \right), \quad (2)$$

where G_{dB} is the receiver gain in dB.

To further reduce noise, $PhDet1(t)$ and $PhDet2(t)$ are acquired from a series of waveforms, with f_{ref} adjusted by amounts on the order of a tenth of a part per million between waveforms to approximately track the resonant frequency, which can drift due to slight variations in temperature [27]. The values for $A_{RF}(t)$ and $\phi(t)$ determined with Equations (1) and (2) from the series of waveforms are averaged, and the acoustic logarithmic decrement δ is determined from a fit of the averaged $A_{RF}(t)$ vs. t .

The instantaneous resonant frequency $f(t)$ during ringdown is determined from the averaged $\phi(t)$ and average drive frequency f_{ref} through the following relation [33]:

$$f(t) = \frac{1}{2\pi} \frac{d\phi(t)}{dt} + f_{ref}. \quad (3)$$

If a specimen has no elastic nonlinearity (i.e., $f(t)$ is constant during ringdown), the slope of $\phi(t)$ vs. t will be constant. An average of $\phi(t)$ determined from multiple waveforms involves an average of the linear contributions to $\phi(t)$ vs. t , and variations in the reference frequencies of the waveforms included in this average have no effect in subsequent analysis of relative nonlinear shifts in resonant frequency determined from changes in $d\phi(t)/dt$ during ringdown [27].

4. Results

Acoustic measurements on specimens NIST5 and NIST6 began at the end of October, 2018, approximately nine months after the specimens were built. The strongest peaks in the resonance spectra of both specimens were found to be in the range of 250 kHz to 350 kHz. A resonance near 270 kHz was selected as the primary focus of measurements, on the basis of its relatively low signal interference from nearby resonances. During the subsequent three months, measurements were performed on the stars in each azimuthal orientation (i.e., with each of the five points of the star vertical). As described in Section 3.1, this report is focused on data acquired with the orientation depicted in Figure 1b (i.e., with the point of the star between R2 and R3 at the top of the specimen).

Figure 3a shows $A_{RF}(t)$ during resonant ringdown of NIST5 and NIST6 after excitation with 4.6 ms tone bursts in January 2019 (solid curves).

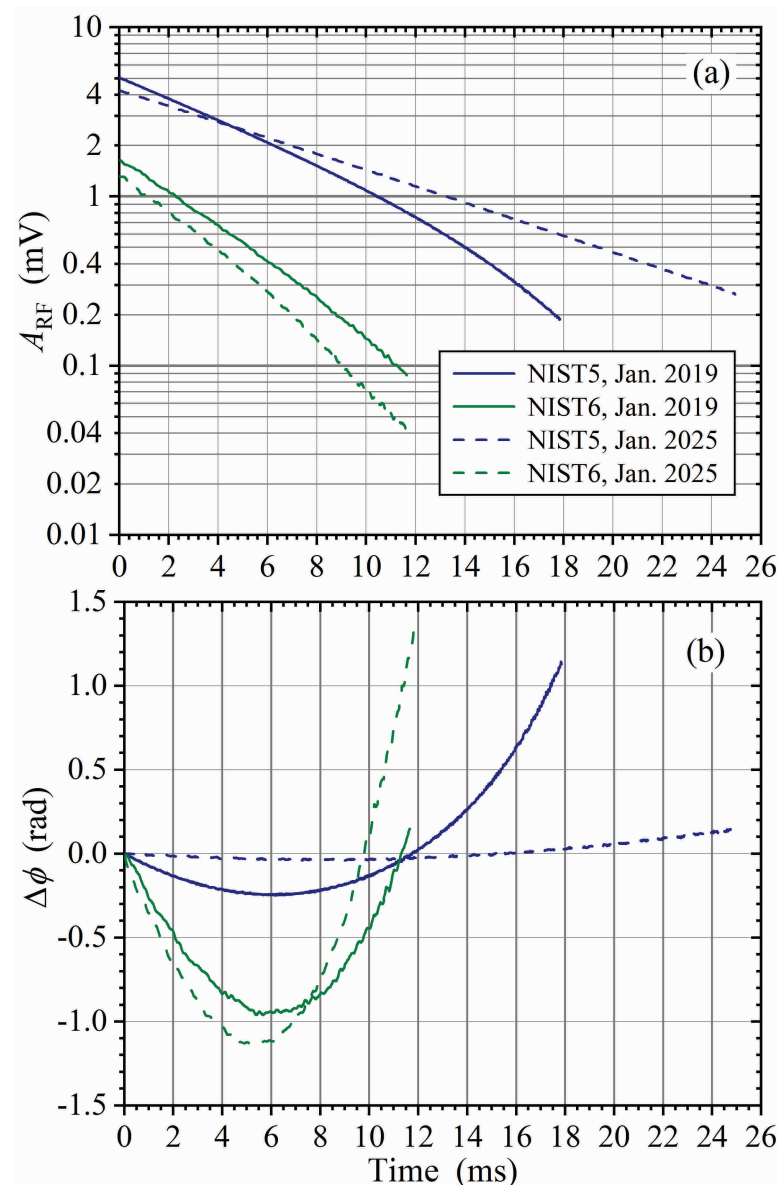


Figure 3. (a) RF amplitude A_{RF} and (b) change in phase $\Delta\phi(t)$ vs. t during ringdown of waveforms from NIST5 and NIST6 in January 2019 and January 2025. $\Delta\phi(t) \equiv \phi(t) - \phi(0)$. Excitation frequencies of NIST5: (270.437 ± 0.001) kHz and (270.407 ± 0.003) kHz in January 2019 and January 2025, respectively. Excitation frequencies of NIST6: (269.575 ± 0.001) kHz and (269.574 ± 0.001) kHz in January 2019 and January 2025, respectively.

Each of these datasets for $A_{RF}(t)$ is fit over the first 8 ms of ringdown to an exponential function of the logarithmic decrement δ , with the frequency during ringdown approximated as constant and equal to the corresponding drive frequency f_{ref} (listed in the caption to Figure 3):

$$A_{RF} = A \exp(-\delta f_{ref} t). \quad (4)$$

Values of δ from these fits are $(5.44 \pm 0.01) \times 10^{-4}$ and $(8.43 \pm 0.02) \times 10^{-4}$ for NIST5 and NIST6, respectively, where the indicated uncertainties include only the standard error of the least-squares analysis. An additional contribution to the uncertainty in δ of similar magnitude arises from variations in resonant frequency during ringdown over the time period of the fit, as described below. The semi-logarithmic plots of A_{RF} vs. t shown in Figure 3a have greater slopes at lower amplitudes, corresponding to greater δ at lower vibrational amplitudes.

Figure 3b shows the change in phase $\Delta\phi(t) (\equiv \phi(t) - \phi(0))$ during ringdown of these waveforms (solid curves). The corresponding time-dependent resonant frequency is determined from these data and Equation (3) with Savitsky–Golay parabolic smoothing in the calculation of $d\phi(t)/dt$. In previous studies, nonlinearity determined from measurements of amplitude dependence of resonant frequencies has typically been quantified as the slope of fractional shifts in frequency vs. vibrational or signal amplitude over a range in which the slope is approximately constant [21,25]. The results in Figure 3a,b lead to no such range of approximately constant slope. The slope continuously decreases with increasing A_{RF} and, as shown in Figure 4 (solid curves), this behavior is so pronounced that fractional shifts in frequency are close to linearly dependent on the logarithm of A_{RF} . In this figure, the fractional shifts in frequency are arbitrarily referenced to the frequency $f_{0.4}$ at $A_{RF} = 0.4$ mV during ringdown. Linear fits of these results for $(f(t) - f_{0.4})/f_{0.4}$ vs. $\log(A_{RF}/\text{mV})$ over the range $0.3 \text{ mV} < A_{RF} < 1.5 \text{ mV}$ yield slopes χ of $(-163.2 \pm 0.8) \times 10^{-6}$ for NIST5 and $(-291.6 \pm 0.8) \times 10^{-6}$ for NIST6, approximately 79% greater for the specimen with designed-in cavities.

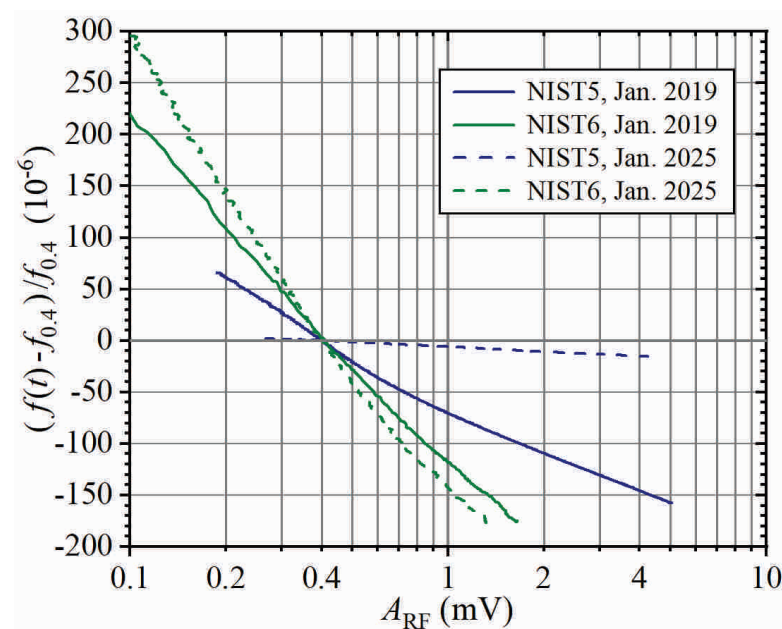


Figure 4. Fractional shift in resonant frequency $f(t)$ of NIST5 and NIST6 as a function of RF amplitude A_{RF} on the coil during ringdown in January 2019 and January 2025. For each specimen, the fractional shifts are referenced to the instantaneous frequency determined at an A_{RF} of 0.4 mV.

As mentioned in Section 1, two previous studies of commercially pure AM aluminum found that nonlinearity and loss decreased substantially with accumulated duration of acoustic excitation over periods of hours [24,25]. Those studies found no evidence for time dependence in the absence of acoustic excitation, and consequently no evidence of thermally activated material changes at ambient temperatures. During the January 2019 measurements of CoCrMo reported here, which were performed before the AM aluminum study, no dependence of nonlinearity and loss on acoustic exposure was anticipated, and a systematic study of potential dependence on acoustic excitation was not performed.

Additional NRS measurements of NIST5 and NIST6 were performed six years later in January 2025 (seven years after the specimens were built) with extended periods of excitation in order to assess potential effects of acoustic exposure on nonlinearity and loss. Figures 3 and 4 show results from the first waveforms acquired in these experiments (dashed curves).

Least-squares fitting of the 2025 data for A_{RF} vs. t in Figure 3 to Equation (4) over the first 8 ms of ringdown yields values of δ of $(3.97 \pm 0.01) \times 10^{-4}$ for NIST5 and $(10.20 \pm 0.02) \times 10^{-4}$ for NIST6. Therefore, these data provide evidence that six years of aging after January 2019 induced a decrease of $\sim 27.8\%$ in δ of NIST5 and an increase of $\sim 17.4\%$ in δ of NIST6.

The change in nonlinearity of NIST5 is more dramatic: a linear fit of $(f(t) - f_{0.4})/f_{0.4}$ vs. $\log(A_{RF}/\text{mV})$ of the 2025 data in Figure 4 over the range $0.3 < \log(A_{RF}/\text{mV}) < 1.5$ provides a slope χ of -15.05 ± 0.01 , which is a factor of 10.8 smaller than the value of χ determined from the NIST5 measurements in 2019. The value of χ determined from the 2025 data for NIST6 in Figure 4 is -348.1 ± 1.7 , which is $\sim 19\%$ greater than the value determined from the 2019 measurements of NIST6 shown in the figure.

The 2025 results shown in Figures 3 and 4 are from the first measurements acquired in that year. To explore potential changes in nonlinearity and loss induced by repeated acoustic excitation, measurements on each specimen were continued for a couple of hours with an excitation duty cycle of 0.2% (4 ms tone bursts, 0.5 Hz repetition rate). Measurements of NIST5 over 7479 s found an increase in δ of $(0.6 \pm 0.1)\%$ and no significant change in χ over this period. Measurements of NIST6 over 6929 s found a $(1.6 \pm 0.1)\%$ increase in δ . As shown in Figure 5, the magnitude of χ of NIST6 decreased by a much greater percentage of $(12.2 \pm 0.7)\%$ from $(348.1 \pm 2) \times 10^{-6}$ to $(305.5 \pm 2) \times 10^{-6}$ over a period of 6929 s. The value of χ at the end of this period is $\sim 5\%$ greater than the value determined for NIST6 in 2019.

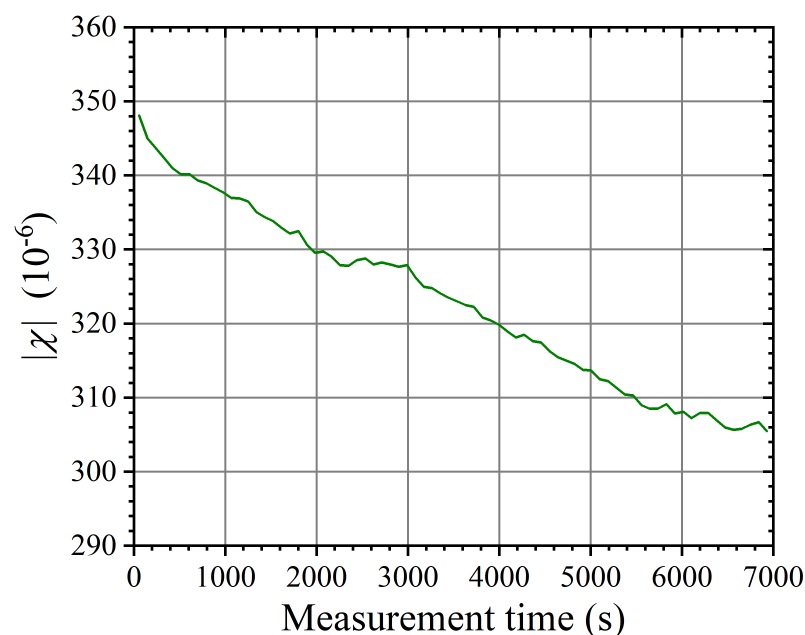


Figure 5. Magnitude of the slope χ of amplitude dependence of fractional frequency shifts $(f(t) - f_{0.4})/f_{0.4}$ of NIST6 (over the range $0.3 \text{ mV} < A_{RF} < 1.5 \text{ mV}$) vs. time since the start of measurements in 2025 with repetitive acoustic excitation: 4 ms burst width, 0.5 Hz repetition rate.

5. Discussion

Table 1 summarizes the results obtained for logarithmic decrement δ and slope χ of fractional frequency shifts vs. $\log(A_{RF})$ in 2019 and the start of measurements in 2025. All of the values for the specimen with intentionally built-in cavities (NIST6) are greater than those of the specimen with no built-in cavities (NIST5). The greater nonlinearity and

loss with built-in cavities is qualitatively similar to previously reported dependence of nonlinearity and loss on low-level porosity in commercially pure AM aluminum [24,25].

Table 1. Summary of results for δ and χ of NIST5 and NIST6 obtained in 2019 and at the start of measurements in 2025.

	2019		2025	
	$\delta \times 10^4$	$\chi \times 10^6$	$\delta \times 10^4$	$\chi \times 10^6$
NIST5	5.44	−163.2	3.97	−15.05
NIST6	8.43	−291.6	10.20	−348.1
NIST6/NIST5 ratio	1.55	1.79	2.57	23.1

As summarized in Section 1, one of our previous studies of AM aluminum [25], which included Ritz calculations of vibrational patterns, Xray-CT, and SEM imaging, concluded that the results were consistent with porosity-dependent nonlinearity and loss arising from lack-of-fusion (LOF) defects generally aligned with the build plane and this LOF contribution being superimposed on porosity-independent nonlinearity and loss from dislocations. This conclusion was based partly on the fact that the porosity dependence of nonlinearity and loss were found to be greater for modes with greater integrated shear gradients across the build plane.

An exploration of hypotheses for dominant physical mechanisms of nonlinearity and loss in the CoCrMo stars is more challenging, partly because of the multiple forms of built-in cavities in NIST6, complex external geometry, and corresponding complexity of vibrational displacement patterns. Detailed information on symmetry dependence of the nonlinearity and loss would require measurements of multiple modes and finite-element modeling of displacement patterns, which are not pursued here. However, one general feature of the excitation symmetry and corresponding excited modes should be noted. As described in Section 3.1, the transduction configuration shown in Figure 2 produces predominantly azimuthal Lorentz forces on a specimen; therefore, shear displacement gradients of the most strongly excited modes are expected to be primarily perpendicular to the five-fold axis of the stars (build direction). With this displacement symmetry, nonlinearity and loss from shear displacement gradients across LOF defects would be expected to be more significant when these defects are tilted relative to the build plane, which is generally a less common orientation in AM materials. However, the presence of mesoscale cavities in NIST6 is expected to introduce greater complexity to the vibrational displacement pattern, including axially varying displacement gradients perpendicular to the build plane. Therefore, greater nonlinearity and loss in NIST6 may be partly attributed to enhanced hysteretic nonlinear effects within LOF defects.

An additional difference between the CoCrMo stars and the previously studied AM aluminum is that NIST6 was intentionally built with unmelted powder in regions R3 and R5 (Section 2). Hysteretic nonlinear surface interactions are expected to occur between these particles as well as between particles and cavity walls. A study by Cabaret et al. [34] demonstrates the presence of such surface interactions in a chain of spherical beads under oscillating shear stress. Low-frequency vibrational measurements of AM specimens with relatively large built-in cavities containing unconsolidated powder show substantial enhancements in amplitude-dependent loss and nonlinearity, and incorporation of such cavities is being pursued by several research groups as an approach for reducing resonant vibrations of AM structural components [35]. In our NRS study of AM steel at NIST [23], we also found a correlation of nonlinearity and loss with volume fraction of untrapped

powder, but interpretation of this correlation is not straightforward because the specimens had other microstructural differences arising from variations in laser hatch spacing. In the future, it would be useful to pursue research on the relative contribution of untrapped powder to ultrasonic nonlinearity and loss in PBF-LB specimens with a range of built-in volume fractions of unconsolidated powder and no differences in laser parameters.

The changes in χ and δ induced after six years of aging of NIST5 and NIST6 at room temperature have different signs (Table 1), and the magnitude of the aging-induced changes in χ of the two specimens are dramatically different. The effects of repetitive acoustic excitation on χ are also much different: a reduction of $\sim 12\%$ in NIST6 (Figure 5) and no significant change in NIST5. These results lead to the conclusion that the dominant mechanisms of nonlinearity and loss in the two specimens are different. As discussed above, unbonded interface effects are expected to be greater in NIST6. Dislocations are also expected to be a significant source of acoustic nonlinearity and loss in the CoCrMo stars, as in previous studies of additively manufactured aluminum [25] and AM stainless steels [20,36,37]. We suggest that they are the dominant contribution in NIST5. These hypotheses about the dominant contributions in NIST5 and NIST6 are similar to a conclusion from our previous study of commercially pure AM aluminum [25] that the lowest-porosity specimens are dominated by dislocation effects and the highest-porosity specimens are dominated by interface effects (see Figure 10, Table 4, and Section 6.4 in that paper).

The reduction in χ of NIST6 induced by repetitive acoustic excitation may be attributed to acoustically induced nanoscale modification of contacting internal surfaces. This explanation is consistent with the absence of a significant similar effect in NIST5, which has fewer internal unbonded interfaces.

In considering potential physical mechanisms responsible for the great changes in nonlinearity and loss of NIST5 over a period of several years, we note that thermally activated diffusion of point defects to dislocations has been recognized for seven decades as a mechanism of pinning of nanoscale dislocation movement that leads to time-dependent reductions in dislocation-related acoustic loss and nonlinearity [38–41]. To explore the hypothesis that such time-dependent pinning of dislocations is responsible for the substantial observed reductions in χ and δ of NIST5 on a time scale of several years, we consider diffusion rates of various point defects in CoCrMo. AM CoCrMo has a metastable face-centered cubic (fcc) crystal structure at room temperature [42], with Co as the primary atomic constituent. The jump rate Γ of each of the alloying elements, namely, Cr and Mo, and impurities in the Co lattice at temperature T are related to the random-walk diffusion coefficient $D(T)$ [43]:

$$\Gamma(T) = \frac{12D(T)}{a^2}, \quad (5)$$

where a is the lattice parameter (0.3537 nm [44]) of Co and

$$D(T) = D_s \exp\left(-\frac{Q}{kT}\right), \quad (6)$$

with Q being the activation energy for diffusion of the solute and k the Boltzmann constant.

Table 2 lists published values of D_s and Q along with corresponding values of Γ (295 K) for Cr, Mo, and impurity elements that are listed in the ASTM F3213 standard [31] as having allowable concentrations in the range of 0.25% to 1%. Interstitial H, which is not mentioned in the ASTM F75 standard, is also included in this table. Most of the diffusion parameters in this table are those calculated by Naghavi et al. [45]. Approximate values for D_s and Q of C are taken from studies of C diffusion in the similar fcc Fe lattice [46,47], while values for N are taken from studies of CoCrMo [48,49] and values for H from a study of CoCrNiW

(44.0% Co, 24.6% Cr, 23.1% Ni, and 4.6% W) [50], since we found no results for diffusion of these elements in pure Co.

Table 2. Published values of D_s and Q for diffusion of solute atoms in fcc Co and corresponding jump rates Γ at 295 K calculated from Equations (5) and (6).

Element	D_s ($10^{-5}\text{m}^2/\text{s}$)	Q (eV)	Γ (Hz)	Refs.
Cr	1.1	2.65	5.6×10^{-31}	[45]
Mo	2.1	2.72	6.9×10^{-32}	[45]
C	2	1.54	9.4×10^{-12}	[46,47]
Fe	1.7	2.64	1.3×10^{-30}	[45]
Mn	2.2	2.89	8.9×10^{-35}	[45]
N	0.116	1.05	1.28×10^{-4}	[48,49]
Ni	0.92	2.91	1.7×10^{-35}	[45]
Si	1.9	2.54	7.4×10^{-29}	[45]
H	0.002	0.294	$1.8 \times 10^{+7}$	[50]

The values of Γ for all of the elements in Table 2, except those for N and H, show that these atoms are frozen in place at room temperature: C is predicted to complete one jump between interstitial sites after three millennia, and other atoms are predicted to take longer than the age of the universe to complete one jump. In contrast, H diffuses very rapidly, with 1.8×10^7 jumps per second, and N is predicted to take 7.8×10^3 s to complete one jump. Therefore, any H present in the specimen after the build in January 2018 would have fully diffused within the specimen during the year before acoustic measurements were performed in 2019, leaving N, which is interstitial in Co [51], as the only element in Table 2 to consider as a potential source of changes in dislocation pinning on a time-scale of several years.

The drift velocity v_d of a point defect towards an edge dislocation at a distance r and an azimuthal angle that provides maximal attractive force is given by [52]:

$$v_d = -\frac{D(T)}{kT} |\nabla U| \quad (7)$$

where $|\nabla U|$ is the magnitude of the gradient of the interaction energy between the point defect and the dislocation, given by

$$|\nabla U| = |\Delta\Omega| \frac{Gb}{3\pi r^2} \frac{(1+\nu)}{(1-\nu)}, \quad (8)$$

where G is the shear modulus, ν is Poisson's ratio, b is the magnitude of the Burger's vector, and $\Delta\Omega$ is the change in volume resulting from the introduction of the point defect in the lattice ("relaxation volume"). Integration of Equation (7) from an initial distance r_0 from a dislocation to a dislocation core radius of b leads to an approximation of the time Δt for a point defect to become a pinning point of the dislocation:

$$\Delta t = \frac{12\pi kT}{\Gamma G b a^2} \frac{(1-\nu)}{(1+\nu)} (r_0^3 - b^3) (|\Delta\Omega| \Gamma)^{-1}. \quad (9)$$

For fcc crystals, Burger's vectors are typically along a close-packed $\langle 110 \rangle$ direction, so that $b = a/\sqrt{2}$. For fcc Co at room temperature, the single-crystal elastic constants are $C_{11} = 225$ GPa, $C_{12} = 160$ GPa, and $C_{44} = 92$ GPa [53]. The corresponding average of the

calculated Hashim and Shtrikman [54,55] upper and lower bounds for G of polycrystalline Co is 78.5 GPa, and ν is 0.31.

For interstitial N, $\Delta\Omega = 4.8 \times 10^{-4} \text{ nm}^3$ (4.3% of the volume per atom, Ω_0 , in the perfect crystal) [51]). From Equation (9), N is estimated to migrate to a dislocation from a distance of approximately 7 nm in six years. If the concentration of N in the specimen were equal to the maximum of 0.25 wt.% allowed by the ASTM F3213 standard, there would be approximately 146 nitrogen interstitials within a radius of 7 nm along each nanometer of dislocation length. Therefore, migration of interstitial N to dislocations appears to be a credible hypothesis for the physical mechanism responsible for the observed reduction in nonlinearity and loss of specimen NIST5 between 2019 and 2025.

Migration of vacancies to dislocations has been shown to be the dominant mechanism for decreasing loss after elastic loading or quenching of pure and alloyed aluminum [40,41] and copper [56,57] with low-amplitude acoustic excitation, and concentrations of vacancies in as-built AM alloys are relatively high because of rapid solidification during the production of powder and subsequent AM build [58]. Naghavi et al. [45] found the jump frequency Γ_v of vacancies in Co to be

$$\Gamma_v(T) = 2 \times 10^{-13} \exp\left(\frac{E_m}{kT}\right), \quad (10)$$

where $E_m = 0.99 \text{ eV}$. This expression gives a jump frequency of $2.4 \times 10^{-4} \text{ Hz}$ at 295 K. $\Delta\Omega$ is reported to be $-5.5 \times 10^{-4} \text{ nm}^3$ (5% of Ω_0) [59,60]. From these values and Equation (9), the distance from which a vacancy can migrate to a dislocation in six years at room temperature is estimated to be $\sim 9 \text{ nm}$. Therefore, migration of vacancies to dislocations is also a credible hypothesis for the physical mechanism responsible for the reduction in nonlinearity and loss of NIST5 between 2019 and 2025. Note that the estimated time for migration to dislocations is much greater than that reported for vacancies in Al because of the much lower value of E_m in Al (0.61 eV to 0.69 eV) [40,41].

The fact that vacancy migration to dislocations has previously been identified as the physical mechanism responsible for decreasing acoustic loss in other alloys after quenching [41,56,57] makes the hypothesis of vacancy migration a particularly compelling explanation for the long-term changes observed in δ and χ of NIST5. To provide definitive evidence supporting or refuting this hypothesis, future studies should perform measurements of time constants of decreasing δ and χ at a series of elevated temperatures in order to determine the migration activation energy [40,41] and compare this experimental value to known values of E_m for the vacancy.

The hypothesis that an increase in point-defect pinning of dislocations is responsible for decreasing χ and δ of NIST5 over a period of six years naturally leads to the conclusion that similar evolution of dislocation pinning must have occurred in NIST6. Therefore, the combination of this hypothesis and the hypothesis that NIST6 is dominated by hysteretic interface nonlinearity suggests that increases in χ and δ observed in NIST6 after six years of aging can be attributed to aging-induced changes in interface effects with opposite sign and greater magnitude than changes associated with increased dislocation pinning.

6. Conclusions

This study has demonstrated that phase-sensitive nonlinear reverberation spectroscopy (NRS) can be effectively performed on AM metal parts with complex geometries to detect differences in acoustic nonlinearity and loss arising from defects. Measurements performed a year after the AM CoCrMo specimens were built found the nonlinearity and loss to be greater in specimen NIST6 with built-in mesoscale cavities and trapped unmelted powder than in specimen NIST5 with no such cavities or powder. This result is qualitatively

similar to the dependence of nonlinearity and loss on porosity previously reported for AM aluminum [24,25].

Greatly different behavior of NIST5 and NIST6 with respect to aging and extended acoustic excitation leads to the conclusion that different physical mechanisms dominate the nonlinearity and loss of these specimens. These mechanisms are hypothesized as hysteretic nonlinearity of dislocations in NIST5 and contacting internal interfaces in NIST6, including contacting surfaces of unconsolidated powder particles and surrounding cavity walls. This conclusion is similar to that previously presented for AM aluminum specimens with low and high levels of measured porosity and associated LOF defects [25].

Calculation of room-temperature migration rates of vacancies and nitrogen interstitials in a stress field of a dislocation support the hypothesis that the observed changes in nonlinearity and loss of NIST5 over six years arise from increased pinning of dislocations. Vacancies are considered to be the most likely candidate for this effect, since they are present in relatively high concentrations after rapid cooling during powder production and the AM build process and have previously been shown to be responsible for time-dependent decreases in loss of quenched conventional metals. The results on aging dependence suggest that, in addition to NDE for quality control immediately after manufacture, NRS could also be useful for nondestructive monitoring of aging-induced changes in mechanical properties associated with dislocation pinning during service of AM alloys.

The results of this limited study of two CoCrMo specimens suggest that two topics would be especially interesting and useful to pursue in future NRS research on AM alloys. The hypothesis of a dominant contribution of hysteretic internal interfaces in NIST6 leads to the suggestion that it would be useful to study the nonlinearity and loss of a set of specimens of an AM alloy with a range of volume fractions of unconsolidated powder and no other intentional microstructural differences. The hypothesis that time-dependent dislocation pinning by vacancies or interstitial nitrogen is responsible for decreases in nonlinearity and loss of NIST5 over a period of several years would be validated or refuted by measurements at elevated temperatures that enable the activation energy of these changes to be determined and compared with known activation energies for migration of these point defects.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/app16167987/s1>, Table S1: Data plotted in Figure 3a. Table S2: Data plotted in Figure 3b. Table S3: Data plotted in Figure 4. Table S4: Data plotted in Figure 5.

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Abbreviations

The following abbreviations are used in this manuscript:

AM	Additively Manufactured
ASTM	American Society for Testing and Materials
CAD	Computer-Aided Design
CT	Computed Tomography

DFT	Density Functional Theory
ISO	International Organization for Standardization
LOF	Lack-of-Fusion
NDE	Non-Destructive Evaluation
NIST	National Institute of Standards and Technology
NRS	Nonlinear Reverberation Spectroscopy

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