

Advanced Facility for Parallel Thermo-Mechanical Simulation and Synchrotron X-Ray Diffraction

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Abstract In recent years, the number of studies involving in situ measurements experienced an impressive growth in different areas of science. This follows from important advances in several areas of technology, involving precise and reliable sample conditioning, very efficient and fast detectors, and better probe sources. This tendency has been accompanied by the development of new installations specialized on in situ measurements, some of which allow studies involving severe, well controlled, and reproducible thermo-mechanical conditions. Although most of the pioneering experiments were made possible by the customization of pre-existing instrumentation, recently, several beamlines have been built or adapted to work with in situ experiments in synchrotron sources. In order to explore some new areas in materials science, we have developed a new installation in Brazil, named XTMS, or X-ray Scattering and Thermo-Mechanical Simulation station, capable of performing in situ diffraction measurements on samples subjected to extreme and complex thermal and/or mechanical conditions, with excellent versatility and reproducibility. The experimental station has been developed and commissioned by the Brazilian Nanotechnology National Laboratory

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(LNNano) team in collaboration with the Brazilian Synchrotron Light Laboratory (LNLS), both located at the Brazilian Center for Energy and Materials Research (CNPEM).

Keywords Synchrotron sources · X-ray scattering · Thermo-mechanical simulation · Gleeble-simulator

1 Introduction

In recent years, the number of studies involving in situ measurements experienced an impressive growth in different areas of science. This follows from important advances in several areas of technology, involving precise and reliable sample conditioning, very efficient and fast detectors, and better probe sources. This tendency has been accompanied by the development of new installations specialized on in situ measurements, some of which allow studies involving severe, well controlled, and reproducible thermo-mechanical conditions. Although impressive developments have been made in electron microscopy and neutron scattering areas, it is still the X-ray scattering field which provides the largest amount of results. This is due to the given availability of high flux synchrotron sources and the considerably simpler instrumentation required, when compared to neutron scattering. In fact, a wide variety of materials science in situ studies using X-ray diffraction have been reported on the literature, covering fields such as phase transformation kinetics [1–4], stress induced crystallographic transformations [5, 6], welding [7], etc. Although most of the pioneering experiments were made possible by the customization of pre-existing instrumentation, recently, several beamlines have been built or adapted to work with in situ experiments in synchrotron sources such as the ESRF, APS [8], SPRing-8 [9, 10] and Bessy [11]. In order to explore some new areas in materials science, we have developed a new installation, named XTMS, or X-ray Scattering and Thermo-Mechanical Simulation station, capable of performing in situ diffraction measurements on samples subjected to extreme and complex thermal and/or mechanical conditions, with excellent versatility and reproducibility. This unique setup opens new research possibilities to explore the interrelationships between stress/strain, temperature, chemical elements partition, and crystallography of both diffusion and shear driven transformations. The experimental station has been developed and commissioned by the Brazilian Nanotechnology National Laboratory (LNNano) team in collaboration with the Brazilian Synchrotron Light Laboratory (LNLS), both located at the Brazilian Center for Energy and Materials Research (CNPEM). Here are presented the constructive details and capabilities of this state of the art experimental station.

2 Installation Overview

The XTMS installation has been engineered to be a comprehensive source of information regarding the tested sample, while it is subjected to thermo-mechanical treatment. It provides the sample status during the whole experiment by registering the actual temperature and uniaxial strain/stress conditions at which it is being submitted, while simultaneously acquiring dilatometry and diffraction data revealing how the sample component phases behave throughout the programmed thermo-mechanical test. This allows a unique insight on the fundamentals of phase transformations and their interrelationships with the crystalline structure and the material macroscopic behavior.

The thermo-mechanical simulation and macroscopic properties are controlled and measured using a custom built physical simulator mounted in a X-ray beamline located at the Brazilian synchrotron light source (Fig. 1). The tested sample is contained within a controlled atmosphere chamber, which is equipped with incident and diffracted X-ray beam windows. A heavy duty goniometer, which can hold one or two dimensional X-ray detectors weighting up to 18 kg is mounted around the thermo-mechanical simulator chamber, with its rotation axis parallel to the simulator's uniaxial force application direction. Both the thermo-mechanical simulator and the goniometer are mounted on independent motorized positioning tables, which allow the goniometer rotation axis to be centered at the point of beam incidence on the sample. The incident beam energy selection, positioning, and focusing are performed using the optics shared with the XRD1 beamline, which is a diffraction beamline at LNLS dedicated to polycrystalline samples. In addition, the XTMS installation counts with dedicated equipment for optimized incident beam control such as X-ray attenuators, high resolution slits, and X-ray beam position meter, which also act as an incident beam intensity counter. Further information on the specific systems that comprise the beamline optics can be found on the following sections.

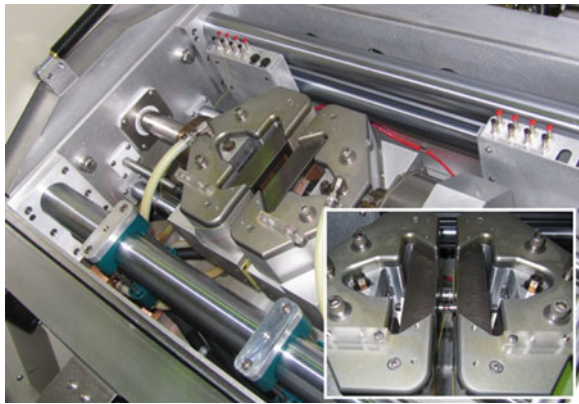
3 Thermo Mechanical Simulator

The thermo mechanical simulator is a custom built GleebleTM system, namely a 3S50TM system, co-developed by the scientific and instrumentation teams from LNNano, LNLS, and Dynamic Systems Inc. (DSI). The physical simulator can control and/or record sample thermal and mechanical history through the experiment, including variables as: programmed and actual temperature, stroke, strain, force and stress. When non contact dilatometry data is acquired, the sample cross section or diameter change with temperature, uniaxial stress and/or time can be recorded to provide an additional insight into the phase transformations undergone by the studied material. The Gleeble thermo mechanical simulator platform was selected due to its well-known flexibility, robust control and penetration on the materials science community.

Fig. 1 XTMS installation at the Brazilian Synchrotron Light Laboratory (LNLS) experiment hall



Fig. 2 Interior of the sample chamber and inset showing the jaws, grips, and the sample free span



Taking advantage of the XTMS potential users expertise on Gleeble simulators, its operational philosophy and sample holders were kept as similar as possible to the commercial systems. Thus, the users' learning curve can be shortened and the intended experiment could be pre-developed and optimized in a conventional Gleeble simulator at LNNano or even at the user facilities. Therefore, in the XTMS simulator the samples are tightly hold between Cu-based or stainless steel grips which are supported on mobile and water-cooled jaws, as shown in Fig. 2. Notice in the figure inset that there is a free span between the grips. The sample is heated by Joule effect while passing a high current (low voltage) through it. At the same time, the sample is being cooled by conduction through the grips and water-cooled jaws. Thus, the control system provides the necessary electrical power to obtain the programmed thermal cycle. When more severe cooling rates are necessary the simulator counts with a Liquid Nitrogen (LN₂) cooling accessory, which has been developed by LNNano. The jaws' movement, which provide the stroke/strain/stress, is powered by hydraulic linear actuators. Unlike conventional simulators where only one jaw moves, because of the necessity to keep the sample

centered with respect to the synchrotron beam, both jaws move symmetrically on the simulator installed at the XTMS station. Temperature and stroke/strain/stress are controlled by a proportional integral derivative (PID) system, where the PID coefficients can be adjusted to optimize the simulator response for specific tests, for example, to provide a faster response, however, with some loss of resolution.

The sample temperature is measured, controlled and recorded using a thermocouple, which is welded to the sample surface at the center of the free span between the grips. The jaws are kept at room temperature by a closed water cooling cycle, which provides a cooling source for the sample. During heating, this causes a temperature gradient along the free span, which is the physically simulated region. Therefore, due to this thermal gradient, only a specific region near to the sample free span center, where the thermocouple has to be positioned, is submitted to the programmed thermo-mechanical cycle.

Typically, for setups with 20 mm free span and programmed peak temperatures around 1,000 °C, the thermal gradient will be less than 5 °C within approximately 3 mm around the central point of the free span. This 3 mm span, where temperature variation can be disregarded for most experiments, is also the typical width of the used X-ray beam. However, if austenitic stainless steel grips and/or larger free spans are used, the temperature gradient along the sample around its middle length will be less severe, and therefore, the temperature variation within the studied region could be much smaller. Nevertheless, in such condition the maximum cooling rates without the use of LN₂ will be compromised.

Most of the grips used to hold and position the samples were developed by LNNano aiming to the correct positioning of the samples regarding the incident X-ray beam. Among these grips can be highlighted ones that allow adjusting the beam incidence angle during the sample setup. Also, several sample designs have been developed and tested by the XTMS developing group. Thus, depending on the experiment requirements in terms of temperature gradient, cooling rates, applied stress or even tested material availability, different designs can be chosen, as seen in Fig. 3.

Heating rates can go up to 500 °C/s, and the maximum achievable temperature is defined by the type of thermocouples used and the sample melting point. The most commonly used thermocouples are types K, R and S, although other types can also be used. Cooling rates as high as -150 °C/s have been achieved with a long free span and the LN₂ cooling system. However, higher cooling rates can be achieved at high temperature ranges, when a small free span is combined with LN₂ cooling. This system also allows well controlled experiments down to subzero temperatures, where -100 °C can be routinely achieved. Measured temperature resolution depends on the range and used thermocouple, but a value of 0.2 °C is typical for a correctly attached thermocouple. Up to four thermocouples can be welded on the sample, normally being used to monitor the temperature gradient on the volume measured by the X-ray beam. A one color pyrometer is also available and can be used for parallel temperature measurements.

Stroke, strain and stress are applied by the symmetric movement of the jaws that hold the grips which tightly hold the sample. A stroke typical resolution of

Fig. 3 Basic Sample designs used in XTMS experiments. Sample length range from 120 to 25 mm, and width from 14 to 5 mm



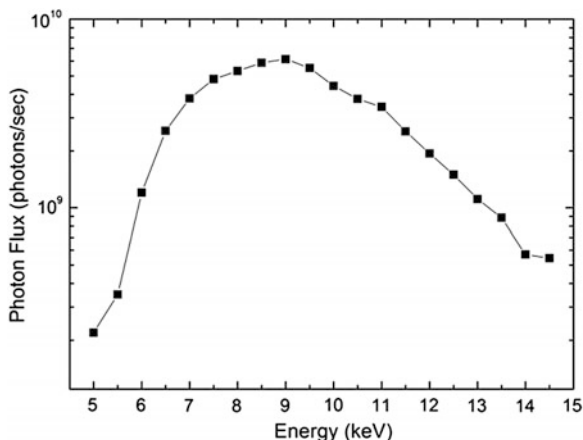
0.01 mm is available. The applied force is measured by a load cell, with 44 kN capability and 0.1 kN resolution. Lower forces load cells can be used, however they are not available at the time. In addition, for very low force values, the resolution will be limited by the system's inherent friction.

Experiments involving stroke/strain/stress are normally performed under tension, whereas, tests requiring elevated accumulated strain may require to be performed under compression mode. However, a methodology for these tests is under development. Strain can be measured/calculated from the sample reduced section length and the jaws stroke assuming that necking is not happening. Unfortunately, this is not case for large strains and/or when the sample is at elevated temperature (due to the thermal gradient). Thus, in such cases, the strain is measured using a non contact laser dilatometer, which is aligned to the center of the free span region, where the peak temperature is located and the thermal cycle is being controlled and recorded. The laser dilatometer provides 10 μm resolution for 2 mm range. Therefore, the strain resolution is defined by the sample geometry and the measuring approach, i.e. jaws stroke or dilatometer. Tests are commonly performed under vacuum, with the lowest achievable pressure being 10^{-3} Torr at the moment. Improvements are being made aiming to reach 10^{-5} Torr. The chamber can also be filled with different gases either for backfilling to reduce oxygen content within the chamber or to run the experiment under a chosen atmosphere, provided that the pressure is below atmospheric pressure. The installation is ready to work with Argon, nevertheless, gas mixtures setup provided by LNLS can be attached to the sample chamber allowing use of artificial air, weld shielding gas mixtures, etc.

4 Beamline

The LNLS Synchrotron is composed of a 1.37 GeV electron storage ring, working with a typical after injection current of 250 mA. The XTMS installation is located at the second hutch of the XRD1 beamline, sharing this beamline with a

Fig. 4 Photon flux at sample position as a function of energy for a storage ring current of 100 mA



high-resolution powder diffractometer. The XRD1 photon source is a 1.67 T bending magnet followed by optics comprised of a Rh coated silicon X-ray mirror and a double bounce Si(111) monochromator. The optics of this beamline is similar to the ones located at other LNLS diffraction beam lines such as XRD2 [12] and XPD. The first mirror provides vertical focalization whereas the second monochromator crystal is used for horizontal focalization and beam positioning. The sample position inside the thermo-mechanical simulator is located 17 m away from the monochromator's second crystal.

Energy resolution at the beamline is of 5 eV at 8 keV. The energy range of the beamline covers from 5 to 14.5 keV. The X-ray beam size at the sample position has a full width at half maximum of 3.6 mm horizontally and 1.2 mm vertically. Focalization can be adjusted to obtain a shorter and wider beam. In addition, the XTMS has motorized slits that allow for smaller beam size selection, at the cost of reduced flux. Figure 4 shows the photon flux at the sample position for the energy range available at the beamline.

5 X-Ray Measurements

Diffraction data is obtained using position sensitive X-ray detectors mounted on a heavy duty Huber goniometer. The detector distance to the sample can be varied from 360 to 600 mm allowing adjustment of the angular resolution of the detector and angular range that can be acquired simultaneously. Two different detector assemblies are available, the first one being a set of two 1 K Mythen linear detector modules, and the second a Rayonix SX165 CCD area detector.

When a flat surface sample design is used, the X-ray beam incidence angle can be selected either by sample design or by using a specially designed grip set, which allows angle setting with 0.1° resolution. However, this angle setting needs to be

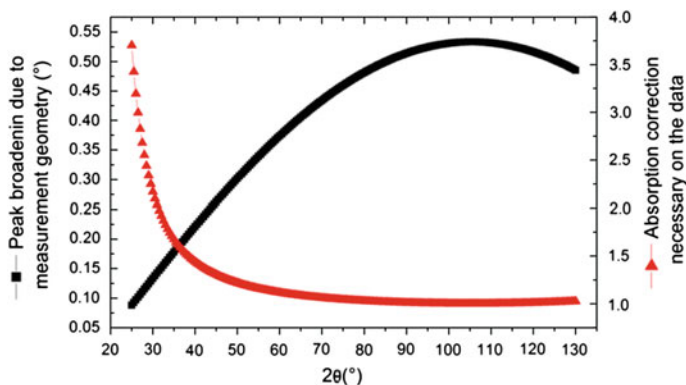


Fig. 5 Peak broadening and absorption correction necessary due to the measurement geometry. Values calculated for 0.5 mm beam height (*vertical direction*) and 15° angle of incidence

predefined and adjusted before starting the experiment, and can only be changed if the termomechanical simulation and X-ray acquisition are interrupted and the sample is removed from the machine. Thus, X-ray diffraction measurements are performed with fixed θ and varying 2θ . Although this geometry is different from the most commonly used $\theta - 2\theta$ scans, it does not greatly affect the data. Major differences are related to peak broadening due to the instrument and the absorption correction variation as a function of 2θ . Figure 5 shows how these two values behave as function of 2θ for commonly used conditions (incidence angle of 15°, 0.5 mm beam height—*vertical direction*). Thus, careful adjustments of these parameters can greatly reduce both effects, but also compromise the incident beam intensity.

Each Mythen 1 K linear detector module is a silicon strip with 1,280 $50 \mu\text{m} \times 8 \text{mm}$ channels distributed in a row. It is a fast detector with 0.3 ms read out time suitable for fast in situ experiments. The detector modules were mounted so that when the detectors are 400 mm away from the sample, their center is perpendicular to X-rays coming out of the sample illuminated area, granting the highest possible 2θ angular range in this sample to detector distance. Each module acquires 9.15° angular range with a 0.5° gap between both modules. The sample to detector distance can also be changed, but further tangent corrections are required. For this assembly, the chamber lid used is a cylindrical window located 280 mm away from the sample. The window covers a 130° angular range, from $2\theta = -5^\circ$ to $2\theta = 125^\circ$.

The Rayonix SX165 is a round CCD area detector with 165 mm diameter active area. It has several binning possibilities allowing a compromise between read out time and resolution. Binning choices range from higher resolution with $39 \mu\text{m}$ pixel size and 5 s readout time to lower resolution with $320 \mu\text{m}$ pixel size and 0,8 s readout time. Although much slower than the linear detectors, the increased detector area provides much higher statistics, being advantageous in

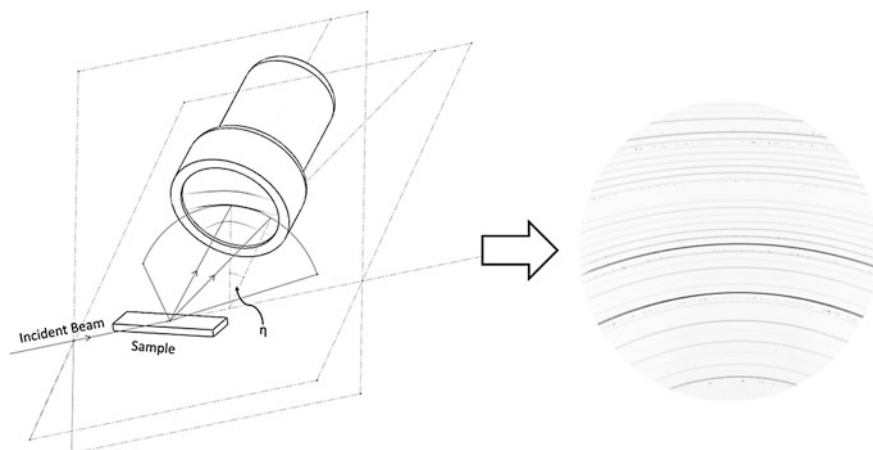


Fig. 6 2D detector measurements schematics. On the inset, an image collected using the detector is displayed. Sample used was Y_2O_3 deliberately contaminated with CeO_2 . The center of the detector was positioned at 30° , and its distance to the sample was 350 mm. Beam energy was 10 keV

reducing effects on the diffraction data caused by texture and low number of probed crystallites. The chamber window for the area detector is a 150×500 mm flat surface allowing measurements from $2\theta = -5^\circ$ to $2\theta = 125^\circ$, and also $\pm 30^\circ$ angular range in the azimuth angle (see Fig. 6).

Measurements can be made taking single acquisitions with one of the detectors fixed in a 2θ position, or scanning the detectors through the desired 2θ range. While the second one gives a much higher amount of crystallographic information, it should be used with care given that the time used to move the goniometer may be too long in terms of the studied phase transformation kinetics. Nevertheless, most of the experiments use combinations of both data collection strategies, where detector scans will be performed to provide enough information for example for Rietveld refinement while the studied phase transformation has not started or has slowed down.

6 Operation

Similarly to the typical programming in a Gleeble[®] thermo-mechanical simulator, experiments are programmed in the XTMS installation by steps, stating the thermal and/or mechanical control variables and their aim values, as well as the other variables that need to be recorded. In addition, for each step it is necessary to state if X-ray diffraction data needs to be recorded and how (scan or single shot). The experiment program is completely specified within SyncSim, a dedicated software developed by the LNNano and LNLS teams for this purpose. Figure 7 shows a graphic representation on how a test would be specified using SyncSim.

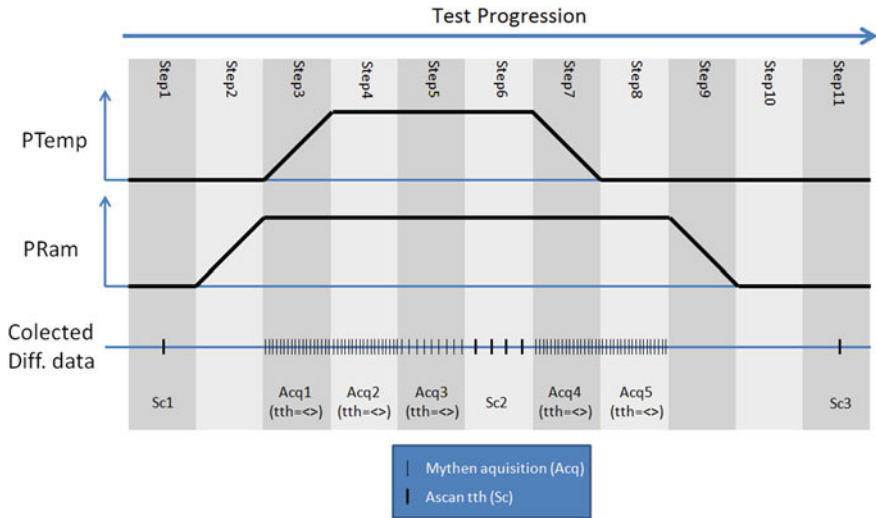


Fig. 7 Example of programmed thermo-mechanical test. PTemp is programmed temperature and PRam can be programmed stroke, strain or stress

7 Applications

As stated previously, the major objective on the designing and construction of the XTMS installation was to build an instrument capable of obtaining high quality data for diverse experiments with different needs without need of major customization. In fact, the installation is ready for experiments requiring high heating or cooling rates, high or low temperatures, long or short temperature dwell times, stress or strain experiments, as well as high acquisition rates or resolution for diffraction data.

In this section we present two experiments performed at the XTMS installation. The first one is a transformation kinetics experiment made on UNS32507 duplex alloy. These materials are formed by a mixture of austenite and ferrite phases, which are stabilized by the careful addition of several alloying elements which, in turn, bring the drawback of potential unwanted phases precipitation, mainly sigma phase (σ), forming at temperatures ranging from 650 to 950 °C [13, 14]. The effect of temperature and time on such phase formation has been widely studied on this and similar materials, including in situ diffraction experiments [15, 16], however, the combined effect of time, temperature and stress on ferrite phase decomposition has not been reported. Using the XTMS installation, time resolved studies on the isothermal decomposition of ferrite (α) in sigma and austenite phases ($\alpha \rightarrow \gamma + \sigma$) have been performed under controlled stress conditions. Samples were heated at a rate of 100 °C/s to a temperature of 850 °C for up to 2 h under uniaxial stresses slightly under the yield strength of these materials at high temperatures. Diffraction data was collected using a linear detector positioned at a

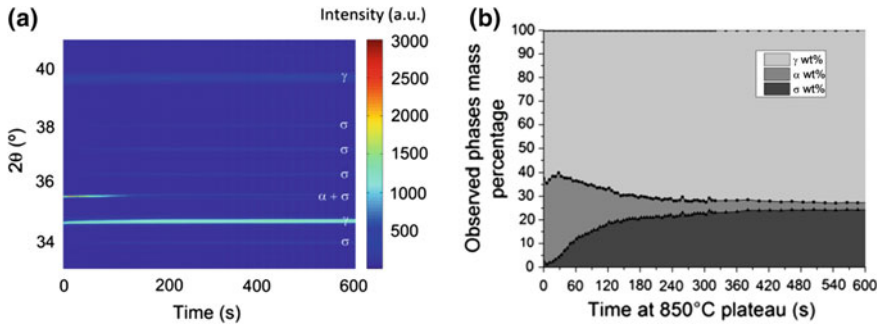
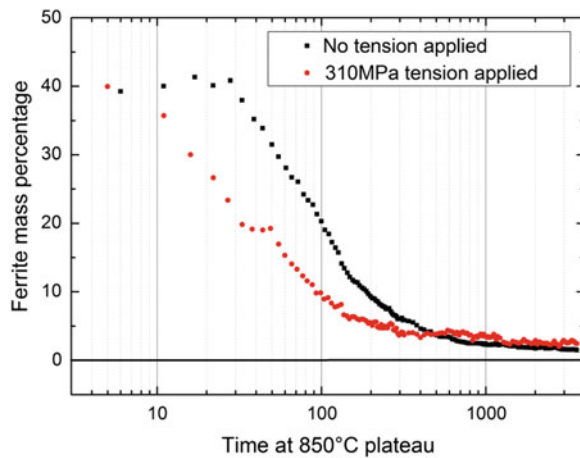


Fig. 8 **a** Measured intensity as a function of the diffraction angle (2θ) and time at 850 °C without load. Peaks are identified as belonging to austenite (γ), ferrite (α), or sigma (σ) phases. **b** Derived mass percentages for the three identified phases as a function of time

Fig. 9 Ferrite phase percentages as a function of time, for a unstressed and a stressed sample



fixed diffraction angle. Figure 8 shows an example of the diffraction data collected. The data was analyzed by determining the peak intensity of the peaks of each phase as a function of time, and accounting structure factor, multiplicity, etc. of each peak, thus determining the phase mass percentage.

In Fig. 9 we show the results for the determined ferrite phase percentage as a function of time. Due to signal-to-noise features, determined mass percentage for ferrite has an uncertainty of 2 %.

The second experiment is an investigation of the austenite behavior during the transformation induced plasticity (TRIP) effect on Supermartensitic Stainless Steels (SMSS). The SMSS typically have high mechanical and corrosion resistance, but these properties are highly dependent on the present phases in the material. These phases can be martensite (M), tempered martensite (M'), delta ferrite (δ) reversed austenite (γ_r) and carbonates. γ_r is a metastable phase, which is

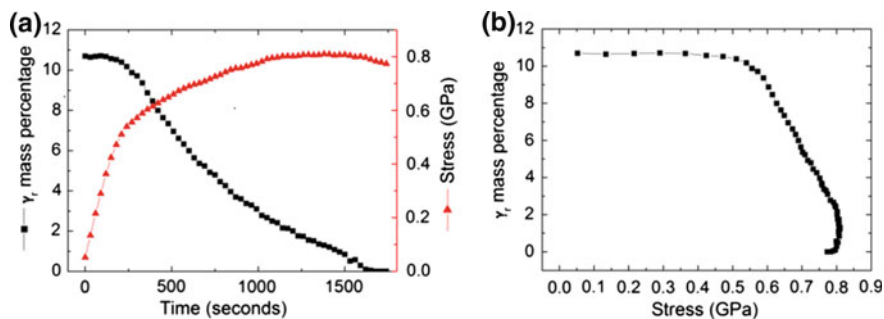


Fig. 10 **a** γ_r mass percentage and stress as a function of time. **b** γ_r mass percentage as a function of stress

stabilized in room temperature by the diffusion of elements such as C, N and Ni during intercritical tempering, in temperatures slightly above the $M \rightarrow \gamma$ transformation start temperature (Ac_1). This is the phase responsible for the TRIP effect on these materials. The existence of γ_r in these materials is known as beneficial, since it increases tenacity and conformability. An example is the effect γ_r has on crack growth, where this phase serves as a barrier to crack propagation. In this experiment the effect of applied tension on the $\gamma_r \rightarrow M$ transformation was observed. This observation has been done before using other in situ techniques such as electron microscopy [17], but measurements of bulk behavior for this material have yet to be reported.

In this experiment, samples containing γ_r were strained at a constant rate of 0.24 %/min, while diffraction data was constantly acquired using a linear detector at a fixed diffraction angle, at a rate of 2 images per minute. Figure 10 shows the results for one sample, which had 10.5 wt.% of γ_r . This sample had been pre-stressed at 0.54 GPa. Figure 10a shows the temporal evolution of stress and the derived γ_r mass percentage whereas Fig. 10b shows the correlation between these two variables.

8 Further Instrumentation Developments

Although XTMS is already open for users from the international community, ongoing developments aim to improve the installation's performance, versatility and the reliability of the acquired data. Some of these developments are listed below.

Improvements on atmosphere control and vacuum aim to achieve pressures under 10^{-5} Torr. An oxymeter will be added to the sample chamber. Finally, a mass spectrometer will be engineered to collect data from the chamber in order to provide quantitative data regarding the atmosphere and sample interaction with it.

While the beam must be carefully aligned with the sample surface, in case of elevated strains this alignment will be lost, causing artifacts in the diffraction data due to sample shift. For this reason a position (height) correction system is being developed. This system will use the non contact laser dilatometer reading to retrofit the simulator positioning table, allowing for the whole simulator vertical position to be corrected as the sample cross section is reduced due to necking.

As mentioned before, the thermo-mechanical simulator and the goniometer are mounted in independent positioning tables, implying that after each new sample is mounted, the thermo-mechanical simulator requires a fine goniometer alignment. Although this is not a time consuming task for experienced synchrotron beamlines users, this is a tedious process, which requires constant user input and that could be challenging for newcomers. The XTMS design and construction philosophy is to attract top notch scientist, preferably with some experience on physical thermo-mechanical simulation, disregarding previous experience with synchrotron beamlines. Therefore, tasks such as sample and beamline alignment should be made as simple as possible for them. Thus, using an optical alignment system already mounted in the goniometer arm, this process will soon be done automatically, with little need of user input.

As well as the above listed improvements, future plans include moving the XTMS installation to a second beamline at LNLS, with higher flux and slightly higher energies (30 keV) and in approximately 4 years to the new Brazilian photon source. In fact, the XTMS' major current limitations are the available photon energy range and photon flux. Typical minimum time resolution for diffraction data is limited to approximately 1 s, and this is restricted only by photon flux if linear detectors are used. In addition, higher energies will allow a bigger gauge volume for the diffraction data, increasing statistics and reducing grain size and surface effects.

The new Brazilian synchrotron source, Sirius, currently under construction, will be a third generation machine with low emittance and high brilliance [18]. XTMS will occupy one of this new machine's beamlines, namely JATOBA, dedicated for High energy Tomography and Laue Diffraction. This will be a Wiggler beamline with photon energy range from 30 keV to 250 keV, 10^{-2} $\Delta E/E$ energy resolution and a $1 \times 1 \mu\text{m}$ beam size, greatly increasing the possible experiments and versatility of the installation.

9 Contributions

Leonardo Wu, Guilherme Faria and Thais Alonso are part of the XTMS development and operation team. They worked together with the other authors in the design and construction of all features and equipment involved in the installation. Augusta Isaac was instrumental on the selection and funding achievement for the 2D detector. James Piton was fundamental for the developed the control software. Regis T. Neuenschwandern is head of the design and instrumentation group at

LNLS, which contributed enormously to the thermo-mechanical simulator customization, positioning systems design and fabrication. Dr. Antonio J. Ramirez is head of the XTMS development and operation team. He idealized the installation and supervised every step in its construction since the start of the project.

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