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Rotatable multifunctional load frame for neutron diffractometers at FRM II – design, specifications and applications

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Abstract

Novel tensile rigs have been designed and manufactured at the research reactor Heinz Maier-Leibnitz (FRM II, Garching near Munich). Besides tensile stress and pressure, also torsion can be applied. The unique Eulerian cradle type design (ω, χ, and φ axis) allows orienting the sample with respect to the scattering vector. Applications of these tensile rigs at our neutron diffractometers enable various investigations regarding to structural changes under mechanical load, e.g. crystallographic texture evolution, stress-induced phase transformations or lattice expansion and the anisotropy of mechanical response.

Keywords: neutron diffractometer, load frame, tensile rig

1. Introduction

The understanding of structure property relationship is an essential condition for the development and improvement of engineering materials. Such investigations often involve *in-situ* characterization of materials under external load. In particular neutron diffraction offers the possibility to follow structural changes on a microscopic level in correlation with mechanical behaviour. In this respect neutron diffractometers are often equipped with load frames to investigate stress-induced phase transformations, lattice strain or crystallographic texture developments [1-5]. In general, neutron diffraction studies give complementary information to X-ray or synchrotron methods. Due to large penetration depths of neutrons even for wavelengths >1 Å, relatively large gauge volumes or bulk samples can be investigated, enabling even coarse-grained and gradient materials to be characterized easily.

At the research reactor FRM II the two neutron diffractometers SPODI and STRESS-SPEC are the instruments of choice for materials characterization [6]. The materials science diffractometer STRESS-SPEC is designed for strain and texture analysis. In addition, this instrument enables a fast collection of powder diffraction patterns, e.g. for the study of reaction kinetics, at a limited scattering angle range. The high-resolution powder diffractometer SPODI is designed for structure refinement on complex systems. Particular attention is paid to enable the characterization of functional materials under special environmental conditions. More details on the design, specifications and applications of the diffractometers SPODI and STRESS-SPEC can be found in the publications, respectively [7-9]. Both instruments provide complementary capabilities for the structural investigations of materials under mechanical load. For these applications two novel, compact load frames were designed for use on both diffractometers.

The development of the load frames was driven by research projects carried out at the neutron diffractometers at FRM II. Moreover, the load frames provide a broad range of applications for materials investigations to the user community. Both frames allow to orient the load axis with respect to the incident beam achieved by a χ-rotation axis. One rig ("unixaial rig") offers uniaxial tension and compression which is optimized for texture analysis (e.g. pole figure measurements) by free sample rotation around the φ –axis. The second rig ("multiaxial rig") enables in addition to tension/compression the application of torsion.

In this contribution, we report on the design and specifications of the new tensile rigs. First results of experiments on nickel titanium shape memory alloys are also presented.

2. Layout, specifications and data acquisition

2.1 Design and specifications

The two load frames are illustrated in figure 1 showing the commonalities as well as their differences. Both rigs are designed for a maximum load of 50 kN in tension or compression and share a common basis structure. They are constructed as double column machines attached to identical rotation frames. This allows to remotely rotate the load axis around 90° from a horizontal to a vertical setup. This rotation is exactly the same as the χ-rotation as used in a standard Eulerian cradle. The χ-tilting of a tensile rig is achieved by a stepper motor and monitored by an optical encoder. The base plate of the rotation frame can be leveled out by a three point fixture as well as positioned in x- and y-directions by adjustment screws.

Tensile axis (for tensile and compressive force) and torsion axis (in case of the multiaxial rig) are equipped with servo motors and load cells or a torque sensor, respectively. The motors are attached to the axis via cam belts. The uniaxial rig allows an additional rotation (φ- rotation) of the sample around the load axis under full load using a stepper motor (figure 2). In case of the multiaxial rig the stepper motor is substituted by a servo motor providing a torque of 100 Nm (figure 3). The load cell of the multiaxial rig is constructional decoupled from the torsion axis so that no torque acts on it.

Both load frames come with a range of grips and additional equipment like furnaces (see chapter 2.3). Their compact design makes them highly portable and enables usage also on other instruments. An overview of the main design specifications are summarized in table 1.

2.2 Control and data acquisition

The load frames are controlled by electronics and software purchased from DOLI Company. Both axes corresponding to tensile stress/compression and torsion can be operated in following modes: constant load or cycling with fixed amplitude. The load increase can be carried out either stepwise or in a continuous mode. In general, continuous loading enables fast data collection and diminishes creep or stress relaxation effects [1]. The axes for stress/compression and torsion can be controlled independently or synchronized. Experiments can be carried out either under load control, position control or strain control mode.

A common interface to the data acquisition system (DAQ) of the diffractometers SPODI and STRESS-SPEC was developed in house so that the tensile rigs can be operated under remote control. Following target values can be set via DAQ: load, torsion, sample extension (i.e. “strain”) or alternatively position of the crossbar (i.e. “position”) as well as the torsion angle. The speed to reach each value can also be set independently.

The axes for χ- and φ-rotation are integrated as standard motors into the instrument control software. Together with the rotation of the main sample table (ω-axis) the sample can be oriented with respect to the incident beam by an Eulerian cradle like movement. The φ-axis supports pole figure measurements either by a step scan (e.g. with a 5×5° grid) or a continuous scan (which can reduce the positioning time considerably [8]). Furthermore the number of detected grains is increased up to a factor of 10 using the continuous scan method, which improves grain statistics.

2.3 Auxiliary Equipment

Grips for tension and compression experiments were designed for standard samples referring to DIN 50125. The specimens shape can be round with maximum diameter of 10 mm or rectangular with a maximum width of 6 mm depending on the material. For torsion experiments a modified sample geometry based on round standard samples was developed to avoid slip of the sample under torque. The macroscopic strain is usually measured by clip-on extensometers. Additionally a camera based system is available for contact-free recording of the sample extension. In order to enable experiments under mechanical load and high temperatures a mirror furnace for the tensile rigs was developed and successfully applied in first experiments up to 1000 °C.

3. Experimental examples

Force and torque sensors of the load frames were calibrated by means of certified sensors. First test experiments using different modes were performed on steel samples [10]. In the following we report on neutron diffraction studies on nickel titanium shape memory alloys.

3.1 *In- situ* structure analysis at SPODI

Experiments at the high-resolution diffractometer SPODI were carried out to study the anisotropy of the mechanical response of nickel titanium shape memory alloys with a nominal composition of 50.14 at.% Ni and monolinic symmetry (space group $P2_1/m$). The multiaxial load frame was set to a fixed ω-orientation of 45° with respect to the incident beam. Diffraction patterns were collected using a wavelength of 2.536 Å at different strain levels up to 4% strain. The load frame was operated in strain control mode to achieve a constant strain level during the data collection. At each strain level, measurements in different χ-orientations were performed. The analysis of the data yielded the changes in d-values and intensities for various Bragg reflections. These results were used to derive elastic constants based on an approach by Popa [11]. The measurements allowed the separation of the anisotropic elastic strain of the material from the anisotropic pseudoplastic strain of the monoclinic martensite phase of NiTi. The pseudoplastic strain was attributed to the adaption of the ferroelastic twin domain structure to the applied stress, which is the key mechanism for the shape memory effect. The ferroelastic twinning/detwinning process was visible from changes in the intensity distribution of the neutron diffraction maxima.

3.2 *In- situ* texture analysis of NiTi at STRESS-SPEC

In-situ texture analysis provides an option to come closer to materials processing (heating, tension, compression, torsion, etc.). Similar activities can also be carried out at synchrotron source. However, the maximum detected gauge volume (~ 1 mm^3) limits its application for the analysis on bulky properties of coarse grained materials. Moreover, the most loading machines are without χ-tilting and φ-rotation which produces two disadvantages. First, only incomplete pole figures were obtained, as reported in paper [13] the pole figure with centre of a 20° non-measured region. This will lead to a lower quality ODF calculation due to the less input data, especially for non-cubic structural materials. Second, under one setup it is impossible to simultaneously obtain intensity, peak position and FWHM pole figures without χ-tilting. One must tilt the sample normal to the beam direction to obtain axial strains. The first application of our load frame to investigate intensity, peak shift and peak broadening pole figures at one measurement was published in reference [10].

Specific example of tensile texture evolution on an identical NiTi is presented. To avoid the miss-orienting of the sample after each tensile strain *in-situ* is strongly required. And for ODF calculation on this monoclinic NiTi large numbers and high quality complete pole figures are also necessary. The measurement was carried out STRESS-SPEC with a wavelength of 1.67 Å using the Si monochromator. The gauge volume was defined by a primary slit of 5 ×5 mm^2 and radial collimator with a field of view of 5 mm. A position sensitive area detector of 300×300 mm^2 covering a diffraction angle 2θ of about 15° at a sample to detector distance of 1040 mm was used to collect the diffracted neutron intensities.

According to previous structure analysis results from experiments carried out on the diffractometer SPODI, pole figures were measured at 0%, 4% and 8% strain, respectively. At each strain value the standard equal angular scan method for complete pole figure measurement was used. The scan involves data collection after a stepwise change of the χ-angle from 0 º to 90º during a complete φ-angle rotation from 0 º to 360º at each χ-position. A scanning routine was implemented [8] to collect all the diffraction patterns while the φ-axis is rotated continuously with a speed of 5° in 90 seconds. A complete pole figure at one detector position took a total measurement time of 10 hours. Three detector positions were needed to obtain 10 reflections of the monoclinic NiTi alloy in order to calculate the orientation distribution function (ODF). Figure 4 shows a 2D image and its summed diffraction pattern at the first detector position at 2θ= 42.4°. The software package StressTextureCalculator (SteCa) [12] was used to extract the pole figures from the measured data.

Figure 5 shows the development of the pole figures with increasing strain of 0%, 4% and 8%, respectively. A radial concentration in the pole figures is observed from which the formation of a fibre texture can be deduced. The maximum intensity remains nearly stable till 8% strain. A gradual rotation of maximum poles / planes with increasing strain is obvious, which most likely arises from twinning deformation.

In addition, the high quality pole figures will enable us to calculate the complete Orientation Distribution Function (ODF) for the monoclinic martensite phase. This is in particular interesting as in monoclinic sample symmetry the calculation of the deformation components has seldom been performed till now and will contribute to our understanding of NiTi Shape Memory Alloys.

4. Summary

Novel tensile rigs were developed and set into operation at diffractometers STRESS-SPEC and SPODI at research reactor FRM II. The load frames allow tension, compression and torsion while the load can be applied in step wise fashion, cyclic mode or continuous mode. The unique Eulerian cradle type design enables to rotate the load axis with respect to the scattering geometry. Despite their high performance, a compact design could be achieved, enabling the use of the tensile rigs on different instruments. The capabilities of the load frames were demonstrated by neutron diffraction studies on nickel titanium shape memory alloys.

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Table 1: Specifications of both tensile rigs

	tensile rig 1 "multiaxial"	tensile rig 2 "uniaxial"
max. force (for tensile stress / pressure)	50 kN	50 kN
max. cyclic loading frequencies	1 Hz	1 Hz
max. torque	100 Nm	-
max. torsion angle	+/- 49°	-
max. cyclic torsion frequencies	0.16 Hz	-
□-tilting axis	0 - 90°	0 - 90°
□ rotation axis	-	0 - 360°
max. travel of crossbar	50 mm	75 mm
width (space between columns)	200 mm	200 mm

Figure Captions

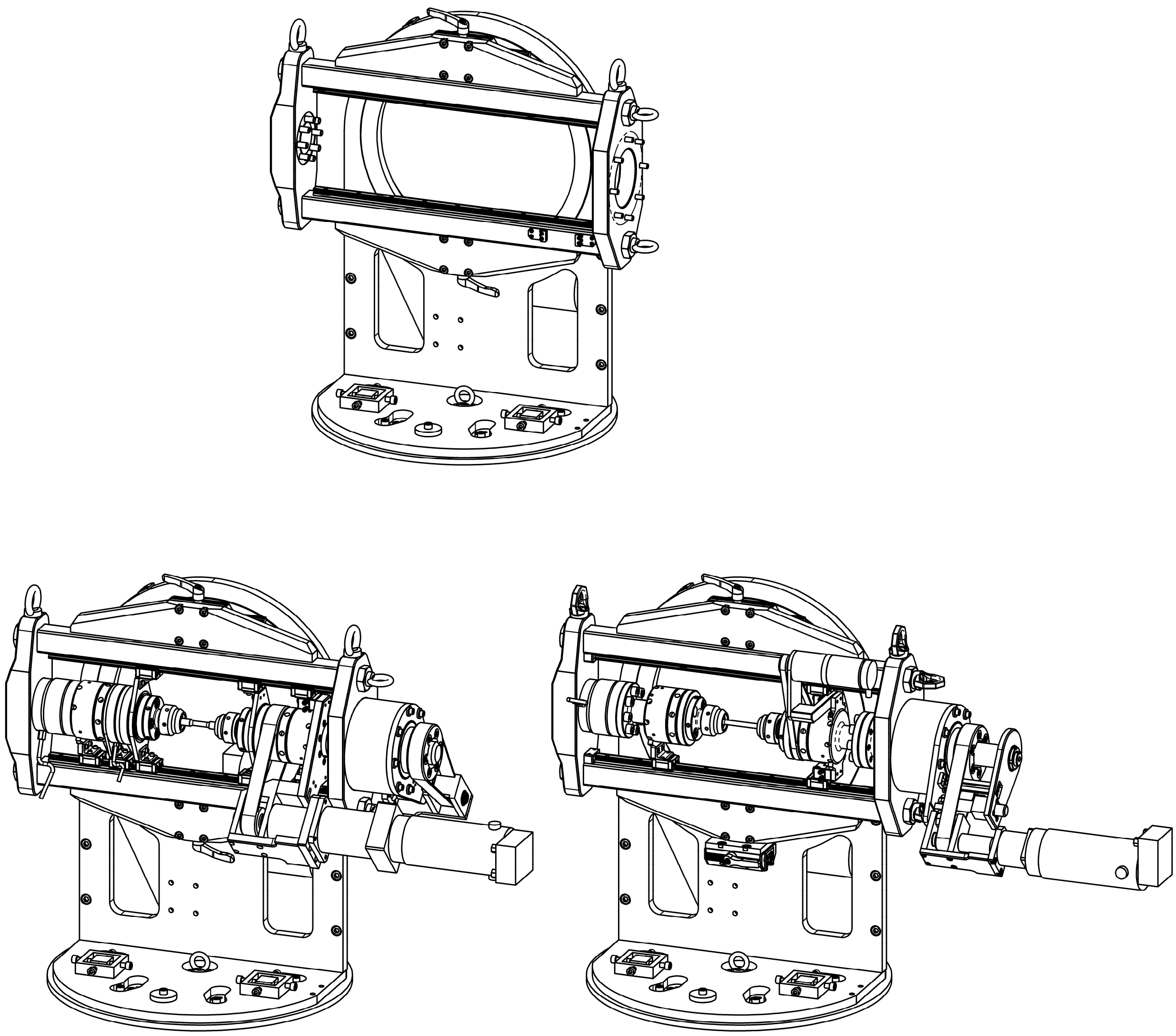
Figure 1: Engineering drawing of the tensile rigs. On the top: the common basis; down left: “mulitaxial version”; down right: “uniaxial version”

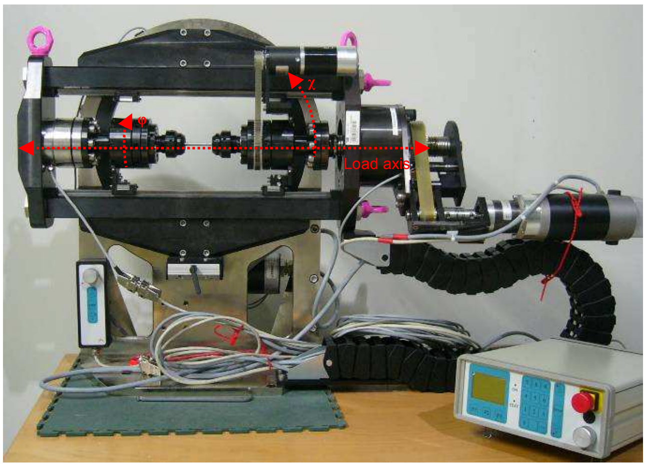
Figure 2: load frame 2 “uniaxial version” with control electronics

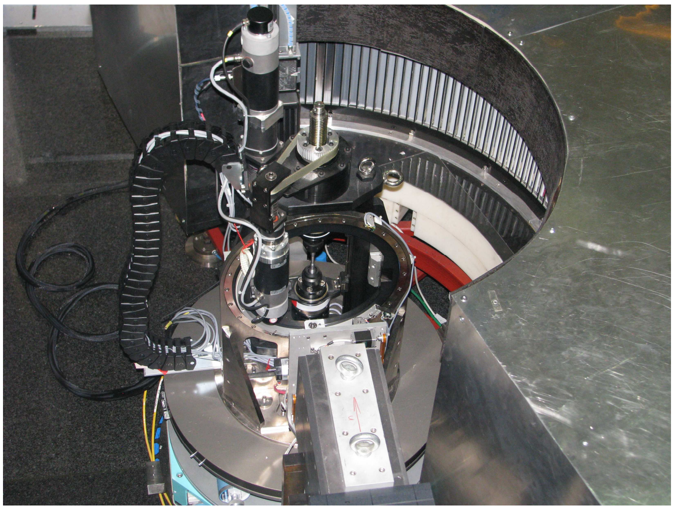
Figure 3: load frame 1 “multiaxial version” on high-resolution diffractometer SPODI

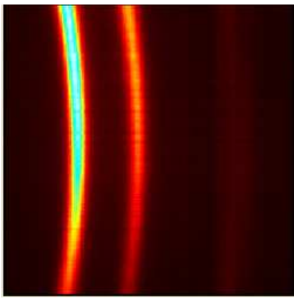
Figure 4: Detector image of NiTi (left) and its sum diffraction pattern (right) including (011), (100) and (-110) reflections.

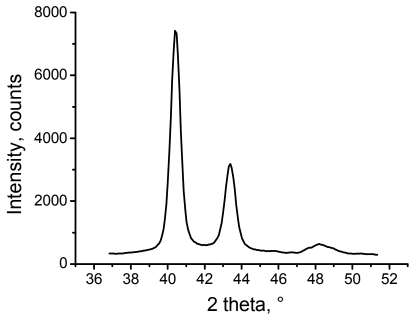
Figure 5: Evolution of (100) and (-110) pole figures from 0% to 8 % strain.




χ
φ
Load axis



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6000
4000
2000
0
Intensity, counts
36
38
40
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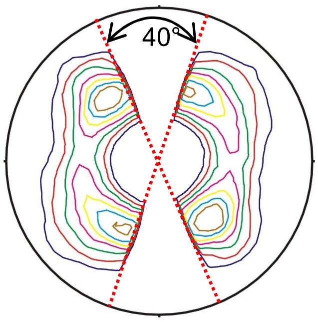
44
46
48
50
52
2 theta, °



Incomplete (100) pole figure obtained using synchrotron