

NEW MAGNETOELASTIC EXPERIMENTS

Roland Grössinger* — Stephan Sorta* — Martin Kriegisch*,**
— Philipp Dessovic*** — Peter Mohn***

New magnetoelastic results performed with standard measuring methods (strain gauge, capacitance dilatometer) are presented. Dynamic measurements of the magnetostriction on undeformed and deformed Ni demonstrates the effect of stress on the magnetostriction. The shape of the $\lambda(H)$ curve change with the frequency of the applied field, indicating the effect of eddy currents. The frequency dependence of the hysteresis losses can be correlated with that of the magnetoelastic losses. Sensitive capacitance dilatometer experiments are used to measure magnetoelastic “de Haas van Alphen” oscillations on polycrystalline Al. This type of experiments shows the coupling of the responsible electrons on the lattice. Thus the determined resonance frequencies correspond to extreme orbits of the electrons on the Fermi surface. The values determined by the magnetoelastic experiment agree well with theoretical data and also with classical “de Haas van Alphen” oscillations performed on Al as well as on a single crystal of PtSn₄.

Keywords: magnetostriction, magnetoelastic losses, magnetoelastic de Haas van Alphen oscillations

1 INTRODUCTION

The magnetostriction is an important intrinsic property of all kinds of magnetically ordered materials. This property is interesting for sensor or actuator applications. On the other side it determines the magnetoelastic energy $\lambda\sigma$ which influences the magnetization process in magnetic materials. The change of length can be between 10^{-8} and 10^{-3} depending on the material. For single crystals the magnetostriction depends on the different crystallographic axes, for polycrystalline material the measured magnetostriction (parallel or perpendicular to the field) is sensitive on deformation, texture etc. For measuring small changes of length applying an external magnetic field various well established experimental methods exist. This paper will show some new experiments which can be done in the area of magnetostriction.

2 STRAIN GAUGE MEASUREMENTS

The most common technique is using strain gauges in combination with a commercial available ac- or dc-bridge. The sensitivity is about 1 ppm. It can be used at low temperatures as well as at elevated temperatures [1]. This method can be applied on all kinds of bulk samples (bars, rings, frames, plates). Additionally dynamic effects can be measured, depending on the bridge.

Generally the magnetostriction is measured in a static field of an electromagnet or a superconducting magnet. On the other side most soft magnetic materials (such as Fe3%Si) are used at 50 or 60 Hz, however the frequency dependence of the magnetostriction $\lambda(H)$ is seldom investigated.

Therefore we measured on a ring shaped (demagnetizing field is zero) sample the frequency dependence of the hysteresis loop and on the same specimen, applying strain gauges, also the magnetostriction as a function of frequency. The material was polycrystalline Ni as well as SPD-deformed (SPD – Severe Plastic Deformation [2]) Ni. After an SPD treatment

(high pressure accompanied by several torque revolutions) the material is in a plastically deformed saturated state. For a polycrystalline, isotropic system equation (1) is valid, however in this case $\lambda_{diff} = \lambda_{sat}$ (“saturation magnetostriction”) with a constant = 2/3. In the case of a deformed sample the constant can be calculated from the degree of texture which is generally a difficult problem

$$\lambda_{diff} = const.(\lambda_{\parallel} - \lambda_{\perp}). \quad (1)$$

For dynamic investigations a 50 kHz bridge (HBM KWS 3085 A) was used. The frequency dependence of the hysteresis loop was measured with a full automatized loop tracer [3]. Before each measurement the sample was demagnetized. Fig. 1 shows the comparison of the magnetostriction of the deformed and undeformed Ni-sample measured with a sinusoidal field at a frequency of 5 Hz [4].

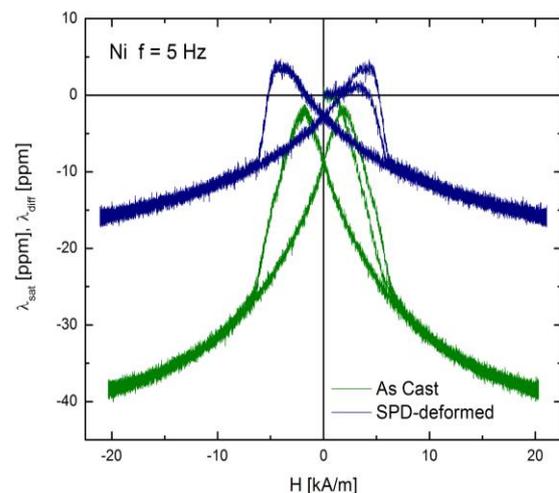


Fig. 1. Strain gauge magnetostriction measurement on a polycrystalline Ni-ring at a sinusoidal field with $f = 5$ Hz including the initial curves

*Institute of Solid State Physics, Vienna University of Technology, A-1040, Vienna, Austria, Wiedner Hauptstr. 8, **Austrian Institute of Technology, Transportation Infrastructure Technology, Vienna, 1210 Austria, Giefinggasse 2, ***Center for Computational Materials Science, Vienna University of Technology, A-1040, Vienna, Austria, Gusshausstrasse 25/134

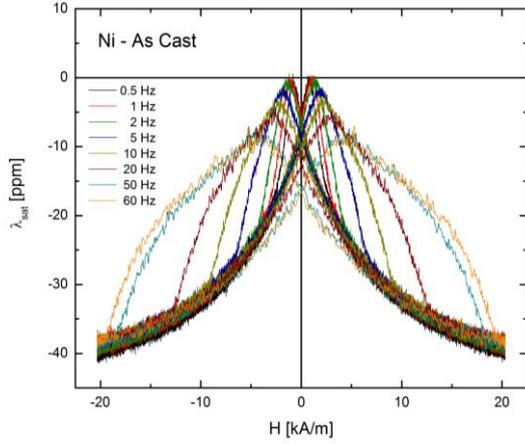


Fig. 2. Strain gauge magnetostriction measurement as a function of frequency performed on the undeformed Ni-ring

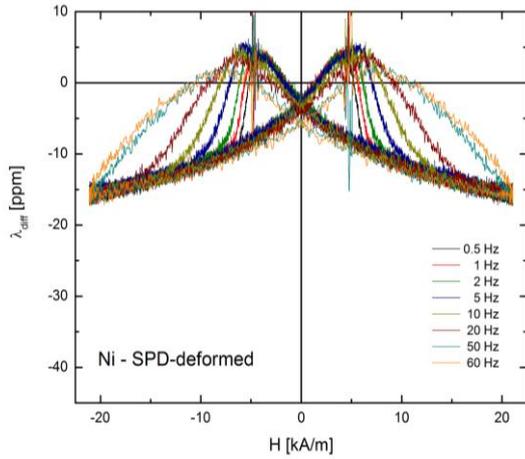


Fig. 3. Strain gauge magnetostriction measurement as a function of frequency performed on the SPD-deformed Ni-ring

The reduction of the $\lambda_{sat}(H)$ curve due to the SPD process is obvious. The effect of this deformation is also visible in the frequency dependence of the magnetostriction – see Fig. 2 and Fig. 3.

With increasing frequency it becomes more and more difficult to approach saturation. This is an eddy current effect. Also the maximum magnetostriction is in the undeformed state about 40 ppm (similar to a literature value [5]), however in the SPD-deformed state it is much smaller. If we regard equation (1) for calculating the magnetostriction one has to consider that the prefactor is unknown for a SPD-deformed material. In order to compare the magnetostriction curves of both samples the same prefactor of 2/3 was chosen. Fig. 4ab shows then the frequency dependence of the hysteresis loops of the undeformed and the SPD-deformed Ni sample.

The total hysteresis losses are $W_{mag} = P_{mag} / f$ which can be generally written as

$$\frac{P_{mag}}{f} = \int_V \frac{d^3r}{V} \int_0^{1/f} \left| \overrightarrow{j}(\vec{r}, t) \right|^2 \rho dt, \quad (2)$$

The total losses depend on the electrical current density \overrightarrow{j} and the specific electrical resistivity ρ and they are calculated over the volume V of the sample. For most soft magnetic materials a loss separation can be assumed [6] dividing into the static losses P_{stat} the classical eddy current losses P_{cl} , and the excess losses P_{exc} :

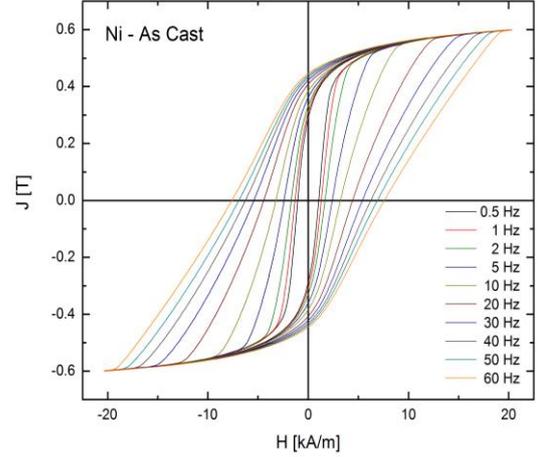


Fig. 4a. Frequency dependence of the hysteresis loop measured on the undeformed Ni-ring

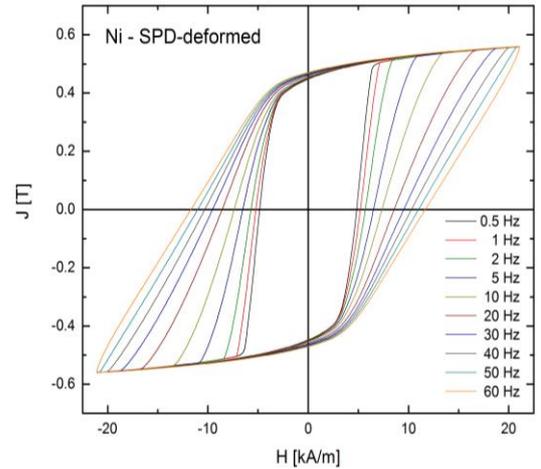


Fig. 4b. Frequency dependence of the hysteresis loop measured on the SPD-deformed Ni-ring

$$P_{mag} = P_{stat} + P_{cl} + P_{exc}. \quad (3)$$

This loss separation can be rewritten as a general function of frequency:

$$\begin{aligned} \frac{P_{mag}}{f} &= W_{mag}(f) = W_{stat} + W_{cl} + W_{exc}, \\ W_{mag}(f) &= C_0 + C_1 f + C_2 f^{1/2}. \end{aligned} \quad (4)$$

Since the area of the hysteresis loops change with frequency and also the hysteresis area in the λ_{sat} versus H curve change systematically, it is interesting to compare these areas as a function of frequency.

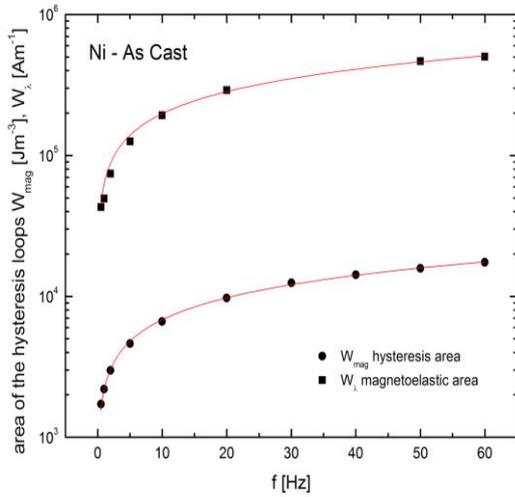


Fig. 5. Dependence of the hysteresis area and the magnetostriction area as a function of frequency for an undeformed Ni-ring

Figure 5 shows the dependence of the hysteresis area and the magnetostriction area as a function of the frequency for the undeformed Ni-sample. Figure 6 shows the same for the SPD deformed material.

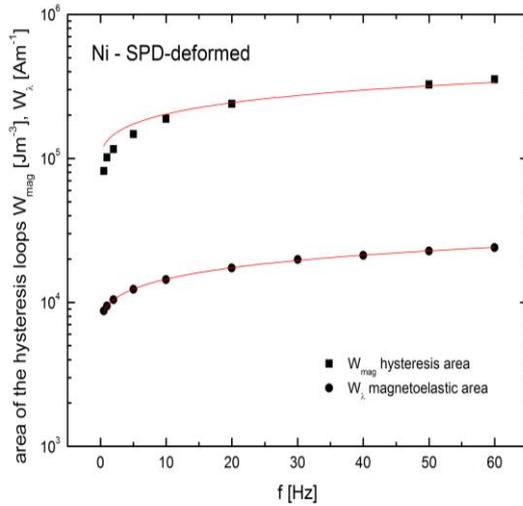


Fig. 6. Dependence of the hysteresis area and the magnetostriction area as a function of frequency for the SPD-deformed Ni-ring

The general character of these curves is similar – therefore a correlation factor may describe this similarity

$$W_{\lambda}(f) = k_E \cdot W_{mag}(f). \quad (5)$$

For the undeformed Ni sample this correlation factor is $k_E^{und} = 29 \text{ m}^2\text{N}^{-1}\text{Am}^{-1}$ delivering very good agreement between these two curves. Also for the SPD-deformed sample a correlation factor can be assumed $k_E^{SPD} = 14 \text{ m}^2\text{N}^{-1}\text{Am}^{-1}$, however here a higher error for low frequencies was found. The difference between these two correlation factors is approximately a factor 2 which corresponds to the magneto-

striction values of these two samples (see Fig. 2 and 3). Considering the different dimensions of the hysteresis area (energy density) and magnetostriction (field - A/m) a physical interpretation of this correlation factor is possible. Depending on the saturation magnetization M_s , the Youngs modulus E respectively the internal stresses σ_i and the magnetostrain ε_{me} have to be considered

$$k_E \propto \Delta \left(\frac{1}{E} \right) M_s \propto \frac{M_s \varepsilon_{me}}{\sigma_i}. \quad (6)$$

Similar investigations were performed on plastically deformed industrial polycrystalline electro-steel (Fe3 %Si) [3] – see Fig. 7. The saturation magnetostriction λ_{sat} can be calculated for an undeformed, isotropic material using equation (1). The constant must be modified to consider an external or internal deformation, which causes a texture.

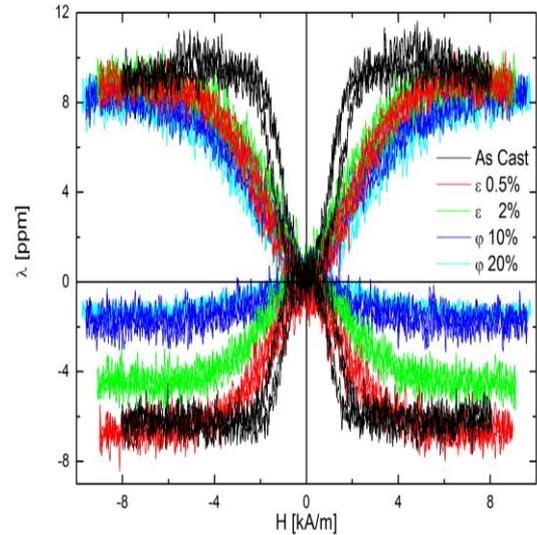


Fig. 7. Longitudinal (up) and transverse (down) magnetostriction at $f = 50 \text{ Hz}$ for the homogeneous deformed electro-steel (Fe3%Si) (0.5%, 2% (by tensile stress), 10% and 20% (by cold rolling) plastically deformed sample) [3]

3 CAPACITANCE DILATOMETER

A sensor for studying magnetostriction and thermal expansion on small samples with a high sensitivity was developed. Since for many new compounds only small samples (1 mm^3) are available and for such investigations a wide range of physical parameters (temperature, field) is necessary, a small and compact dilatometer for a wide temperature range and high magnetic fields based on the tilted plate capacitive dilatometers was constructed [7]. Fig. 8 shows a drawing of the basic construction of the dilatometer.

The dilatometer was calibrated using the thermal expansion data from pure silver [8]. The change of capacity has been measured by an “Andeen Hagerling 2500 A 1 kHz Ultraprecision Capacity Bridge”. A common value for the gap of the capacitance is 0.18 mm, which results in a capacity of

approximately 4 pF. The sensitivity of this system is better than 0.01 ppm – this depends only on the thermal stability of the magnetostriction measurement set-up. Since this cell is constructed from pure silver, there is no parasitic magnetic field influence (magnetostriction) in the measurement signal.

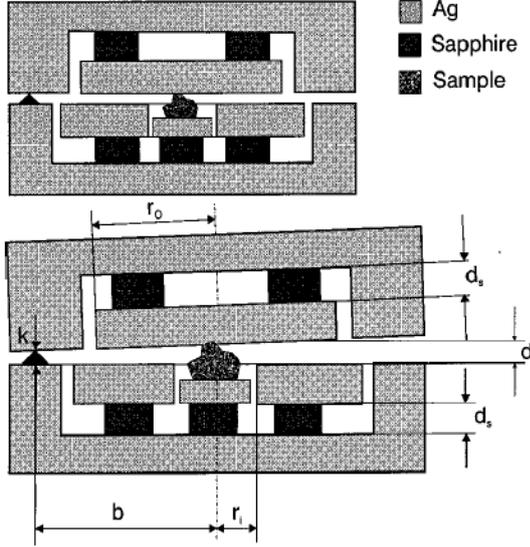


Fig. 8. Schematic drawing of the capacitance dilatometer [7]

In order to demonstrate the possibilities of this dilatometer magnetoelastic “de Haas-van Alphen” (dHvA) oscillations on pure polycrystalline Al (4N) were measured. The de Haas-van Alphen oscillations are periodic to the inverse magnetic field $1/H$ with its frequency proportional to the extremal cross section of the Fermi surface. With the help of the dHvA oscillations it is possible to study the Fermi surface of a metal and gain new insights of its band structure.

Figure 9 shows the magnetoelastic dHvA oscillations measured at $T = 4.2$ K applying a magnetic field up to 9 T. The inset shows the Fourier transform of the measured magnetostriction signal. According to literature [9,10] the periods of the α_1 , α_2 and α_3 orbits of the third-zone Fermi surface were determined to be 27.5 T to 28.2 T, while the β orbit was found to be 46.9 T.

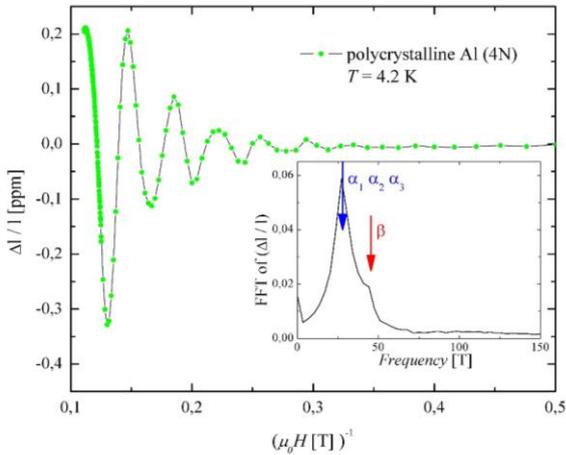


Fig. 9. Magnetostriction measurements versus inverse magnetic field of polycrystalline Al applying a magnetic field up to 9 T - the inset shows the Fourier transform of the measured signal.

Our measurements on polycrystalline aluminium sample are in very good agreement with the literature (as indicated by the arrows in Fig. 9). The rather broad peak in frequency for α_1 , α_2 and α_3 comes from an averaging over all crystalline directions, which is accordance with the angular variation of the dHvA oscillations. See also the de Haas van Alphen orbits for Al as shown in Fig. 10 [10]

These data were also analysed theoretically (using Vienna Ab initio Simulation Package (VASP)) and compared with the experimental data. The most recent “de Haas van Alphen” study of Al were performed by Larson and Gordon [10]. Their results for the (100) direction are given in Table 1.

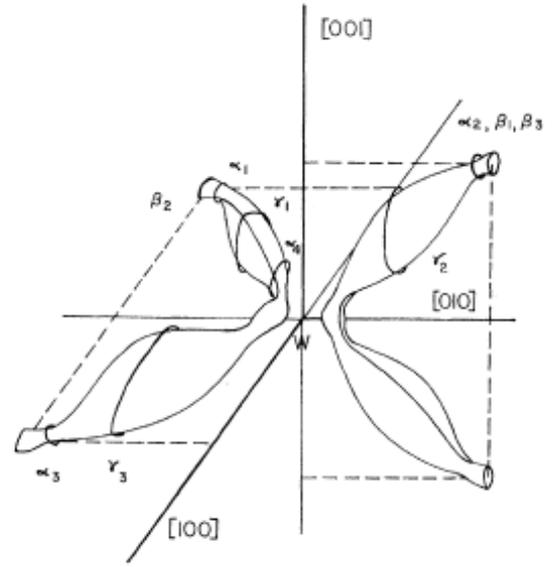


Fig. 10. de Haas van Alphen orbits for Al [10]

Table 1. Orbits, DFT-calculated (Density Functional Theory) and experimental data (Larson et al [10]) compared with magnetoelastic experiment determined oscillations - the B -Field was always applied parallel to $\langle 100 \rangle$.

| Orbit | Calc ν (T) DFT | Exp ν (T) [10] | $1/\Delta B^{-1}$ | FFT |
|----------------|-----------------------|-----------------------|-------------------|----------|
| α_{123} | 29 | 28.2 | 28.7 27.9 | 26 |
| β | 47 | 46.9 | | 43 45 |
| α_4 | 91 196 | | | |
| γ | 405 5468 | 389.1 | 392.1 | |

The α_{123} orbit at 29 T fits quite well with the experimental value. For the β orbit at 47 T only a small indication was found in the Fourier transform of the magnetostriction measurement. The α_4 orbit at 90.8 T was not measured experimentally at all. The 169 T maximum is above/below the α_4 orbit (minimum) at the joining of the arcs next to point W. The orbit at 405 T was measured by both experiments at slightly lower values. There might be some interference in the magnetostriction measurement at the high frequency os-

cillations, seen in the inset at Fig. 9. Since the small sampling rate (determined by the long integration time of the capacitance bridge) the high frequency predictions of the Fourier transform analysis is unreliable.

Magnetostriction measurements were also performed on a single crystal of PtSn₄ at 4.2 K as shown in Fig. 11 [11]. These experiments were compared with ab-initio simulations of the Fermi surface and showed excellent agreement with experiment, which confirms magnetostriction measurements as a powerful tool for measuring dHvA quantum oscillations.

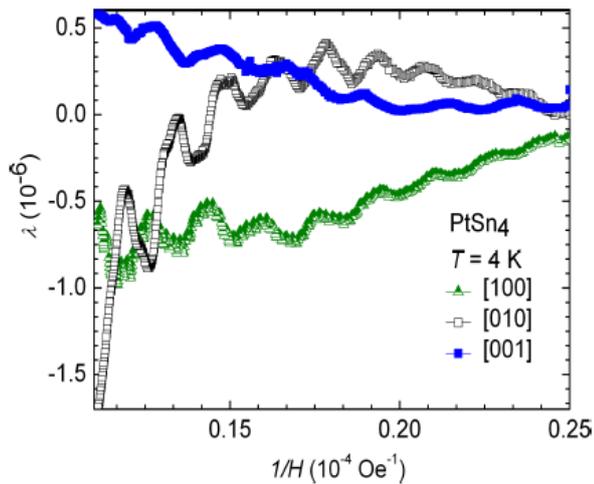


Fig. 11. Change in linear unit cell parameter ($\lambda = \Delta l / l$) of PtSn₄ with changing magnetic field along the three crystallographic directions - the small amplitude of $\lambda(1/H)$ is attributed to the diamagnetic nature of the sample [11].

4 CONCLUSIONS

Dynamic magnetostriction measurements on soft magnetic materials are presented. The effect of residual stress caused by plastic deformation on the $\lambda(H)$ curve is good visible and can be explained by a stress induced texture. A basic problem is here the stress induced rearrangement of the domains which affects the absolute value of the magnetostriction as calculated by $\lambda_{diff} = const \cdot (\lambda_{\parallel} - \lambda_{\perp})$.

The losses as determined from frequency dependent hysteresis measurements can be correlated with the hysteresis area as obtained from magnetostriction measurements. As proportionality factor an expression containing the Youngs modulus E respectively the internal stresses σ_i was obtained.

Quantum oscillations on a polycrystalline sample of Al are shown by measuring the magnetostriction with a high sensitivity in fields up to 9 T. The magnetoelastic oscillations agree well with classical dHvA results as well as with theoretical calculations. Similar oscillations were observed in a PtSn₄ single crystal. Also here the data agree with classical dHvA magnetization measurements as well as with theoretical calculations.

REFERENCES

- [1] J. E. Goldman, *Magnetostriction of annealed and cold worked nickel rods*, Phys. Rev. **72**(6), 529-530 (1947).
- [2] M. Zehetbauer, R. Grössinger, H. Krenn, M. Krystian, R. Pippan, P. Rogl, T. Waitz, R. Würschum, *Bulk Nanostructured Functional Materials by Severe Plastic Deformation*, Adv. Eng. Mat. **12**(8), 692-700 (2010).
- [3] Stefan Hartl, *Magnetic properties of deformed Electrical Steel*, Master-thesis, TU Vienna (2014).
- [4] Stephan Sorta, *Magnetostruktionsmessungen bei Raumtemperatur*, Master-thesis, TU Vienna (2014).
- [5] E. W. Lee, *Magnetostriction and magnetomechanical effects*, Rep. Prog. Phys. **18**(1), 184-229 (1955).
- [6] G. Bertotti, *Hysteresis in Magnetism: For Physicists, Materials Scientists, and Engineers*, Academic Press, San Diego (1998).
- [7] M. Rotter, H. Müller, E. Gratz, M. Doerr, M. Loewenhaupt; *A miniature capacitance dilatometer for thermal expansion and magnetostriction*, Rev. Sci. Instrum. **69**(7), 2742-2746 (1998).
- [8] G. K. White, J. G. Collins, *Thermal expansion of copper, silver, and gold at low temperatures*, J. Low Temp. Phys. **7**(1-2), 43-75 (1972).
- [9] E. M. Gunnerson, *The de Haas - van Alphen Effect in Aluminium*, Philos. Trans. R. Soc. London, Ser. A **249**(965), 299-320 (1957).
- [10] C. O. Larson, W. L. Gordon, *Low-Field de Haas - van Alphen Study of the Fermi Surface of Aluminum*, Phys. Rev. **156**(3), 703-715 (1967).
- [11] M. Inamdar, M. Kriegisch, L. Shafeek, A. Sidorenko, H. Müller, A. Prokofiev, P. Blaha, S. Paschen, *Quantum oscillations in ultra pure PtSn4*, Solid State Phenom. **194**, 88-91 (2013).

Received 30 November 2015