Review articles

# Pressure-tuning of the electronic and magnetic properties of $EuPt_2Si_2$

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## ARTICLE INFO

## ABSTRACT

Using the element and orbital selectivity of x-ray absorption spectroscopy at Eu and Pt $L_3$ edges we investigate the effects of the lattice contraction, induced by temperature and external pressure, on the magnetic and electronic properties of the $EuPt_2Si_2$ system. Our findings point to a clear relationship between the volume and the Eu valence in this material. From XANES experiments as a function of pressure we show that the Europium valence tends to stabilize at 3+ for pressures up to 27 GPa. The XMCD results demonstrate that pressure induced valence change of the Europium ion leads to a suppression of the magnetic ordering of the material. Altogether our results provide direct evidence that Eu-$4f$/$5d$ electronic hybridization effects underlie the mechanism that regulates the valence and magnetic ordering in this material.

## 1. Introduction

Among the rare-earth intermetallic compounds that crystallize in the tetragonal structure, those which contain Eu have attracted special interest because their electronic configuration is sensitive to chemical substitution and applied pressure. The Eu ions can adopt two valences states, namely $Eu^{2+}$ (S = 7/2, L = 0) carrying a high total angular momentum (J = 7/2) and $Eu^{3+}$ with a non-magnetic ground state (S = 3, L = 3, J = L-S = 0). Many factors affect the Eu valence [1–8], such as the local environment which determines the crystal field splitting, the electronegativity and the concentration of alloying partners as well as external parameters like temperature, pressure and magnetic field [9,10]. Moreover, in a number of europium compounds one observes a continuous evolution from a divalent state at high-temperature to a trivalent state at low-temperature with an extended valence fluctuating regime in between. It is well known that systems with unstable $4f$-shells exhibit emergent physical phenomena, such as Kondo effect, electron mass enhancement, valence fluctuations, unconventional (magnetically driven) superconductivity, non-Fermi liquid state, or critical fluctuations in the vicinity of a quantum critical point [5,7,10–16].

The $EuPt_2Si_2$ compound crystallizes in the tetragonal $CaBe_2Ge_2$ type structure, and becomes antiferromagnetic (AF) below $T_N$ = 16 K, presenting a Curie Weiss susceptibility behavior with an effective

moment close to that expected for a $Eu^{2+}$ ($4f^7$) configuration [17–19]. However, in contrast from what is observed in other antiferromagnetic europium divalent compounds [15,16,20–22], Mössbauer spectroscopy studies at ambient pressure reveal that when the temperature is reduced the Eu valence shifts towards a $Eu^{3+}$($4f^6$) state [23]. In addition, Mitsuda et al. [17,24] reported an unusual behavior of the electrical resistivity as a function of temperature, with the resistivity increasing below 120 K and presenting saturation tendency at low temperatures. Such behavior of the resistivity could not be explained based on a pure $Eu^{2+}$ electronic configuration. Moreover, the substitution of Pt by Ni (chemical pressure), even in small amounts (<10%), is able to induce dramatic changes in valence and magnetic ordering in this material [18]. Although chemical substitution is useful to study the relation between lattice parameters and other physical properties, it also leads to an increase in disorder, which can thwart direct interpretations of new physical phenomena. The use of external pressure to continuously tune the ground-state properties of intermetallic compounds appears as an excellent alternative to overcome the disorder problem induced by chemical substitution [25–27]. For instance, Mitsuda et al. [17] reported that pressure induces a collapse of the magnetic ordering and leads to a dramatic change in the residual resistivity ratio (RRR). The collapse of magnetism might be related to valence changes in the Europium ion, while the change in RRR indicate that the scattering

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factors are being modified [24]. However, the interpretation about the link between pressure changes and transport properties as well as the Eu valence remains elusive due to the lack of accurate and direct probe of Eu valence. These open questions make $EuPt_2Si_2$ an ideal candidate to study the correlation between the pressure induced destabilization of the Eu valence and the electronic and magnetic properties of the material.

In order to elucidate the origin of these behaviors and to understand the mechanism that regulates the electronic and magnetic properties of this compound, we exploited the element and orbital selectivity of X-ray Absorption Spectroscopy (XAS) to probe the spin-polarized electronic structure of the $5d$ band states as the lattice is contracted. The concomitant changes in valence and the ordered moments were probed by XANES (X-ray Absorption Near Edge Structure) and XMCD (X-ray Magnetic Circular Dichroism) measurements, respectively. Our findings put in evidence the instability of the Europium valence in this compound as well as show a relationship between the Europium valence and the lattice parameters. The complete spectroscopic description of the electronic structure and magnetic changes under pressure should guide efforts in understanding how valence and magnetic exchange interactions are regulated in similar rare earth intermetallic systems.

## 2. Methods

The $EuPt_2Si_2$ single crystals have been grown by a flux method using Sn as melting flux in a 1:20 ratio compound-formula-unit/Sn. This growth technique is particularly interesting for Europium compound due the Europium high vapor pressure. Pure materials (Eu-99.9%, Pt-99.99%, Si-99.99% and Sn-99.99%) were placed inside an alumina crucible and sealed in quartz ampoule with Argon. The ampoules were taken to a furnace and the temperature was raised to 950 °C at a 20 °C/min rate and remained at that temperature for 48 h. A slow rate of 0.1 °C/min was used to cool the samples down to 400 °C. The samples were subsequently centrifuged to separate the Sn flux and were further mechanically cleaned to remove any excess of Sn. This process provided plate-like crystals typically with $1 \times 1 \times 0.5$ mm dimensions. We used a commercial (Quantum Design) Superconducting Quantum Interference Device (SQUID) magnetometer to characterize at ambient pressure the magnetic properties of our material at different applied fields. The results obtained at ambient pressure are in good agreement with previous reports [17,24] and confirms that our sample crystallizes in a $CaBe_2Ge_2$-type structure with lattice parameters of a = 4.111 Å, c = 10.248 Å, and antiferromagnetic ordering below $T_N$ = 16 K.

X-ray powder diffraction (XRD) measurements as a function of temperature were carried out at the Brazilian Center for Research in Physics (CBPF) in a conventional diffractometer (X'Pert-Pro - Panalytical) using Cu K-alpha radiation and the lattice parameters were determined by Rietveld refinement. High pressure powder XRD were performed at W09A-XDS beam line of the Brazilian Synchrotron Light Laboratory (LNLS) (P < 10 GPa) [28] and 16BM-D beam line (10 GPa < P < 30 GPa) of the Advanced Photon Source (APS). The XRD patterns were collected in transmission mode with an area detector (RAYONYX-at LNLS and MAR345 at APS). The two-dimensional (2D) images were integrated to provide intensity as a function of $2\theta$ using the FIT2D software [29]. Hydrostatic pressure was generated using diamond anvil cells (DAC) assembled with diamond of 300 μm (APS) or 600 μm (LNLS) culet diameters. For the experiments performed at APS (LNLS) we used Helium (4:1 methanol–ethanol mixture) as pressure medium. Due to the DAC limited angular scattering range (25° of scattering angle $2\theta$), the X-ray energy was tuned to 25 keV (LNLS) and 29.2 keV (APS) in order to detect a significant number of Bragg peaks within this angular range.

XAS experiments at ambient pressure at Eu and Pt $L_3$ edges were performed at D08B-XAFS-2 beamline of LNLS. At 4ID-D beamline of APS we carried out in transmission geometry XANES and XMCD measurements at Eu and Pt $L_3$. To generate the circular polarization needed for the XMCD experiment we use phase-retarding optics [30] available at 4ID-D. The XMCD spectra were obtained from the difference ($\mu^+ - \mu^-$), where $\mu^{+,-}$ are the helicity-dependent absorption coefficient normalized by the absorption edge jump, while the spin-averaged XANES is defined as ($\mu^+ + \mu^-$)/2. We note that to check for systematic errors in XMCD signals we carried out XMCD measurements for two directions of the applied magnetic field, along and opposite the incident photon wave vector. A He-flow cryostat able to reach temperatures as low as 5 was inserted into the bore of a split-coil superconducting magnet capable of reaching $\pm 4$ T magnetic field. Although, at ambient pressure, $EuPt_2Si_2$ is antiferromagnetic material, the 4 T applied magnetic field is high enough to induce a spin canting and a net magnetization to be probed by XMCD. To reach pressures as high as 30 GPa we used a non-magnetic, miniature diamond anvil cell (mini-DAC) manufactured by D'anvils loaded with partially perforated diamond anvils in an opposing configuration to a mini-anvil with 300 μm culets. The total diamond thickness in the X-ray's path was around 1 mm, minimizing X-ray attenuation and enabling the measurements at the relatively low energy of Eu $L_3$ edge (6.97 keV). Si-oil was used as pressure transmitting medium. The pressure was applied at ambient temperature and calibrated using the ruby luminescence method [31]. When cooled down to low temperature no change in pressure was detected within a $\pm 1$ GPa error bar, as determined by independent X-ray absorption fine structure (XAFS) measurements on Cu powders [32]. We note that we used different pressure media in X-ray diffraction and X-ray absorption experiment due experimental limitation, however the difference in the hydrostatic conditions between silicon oil (XAFS experiments) and the methanol:ethanol mixtures (X-ray diffraction experiments) will not have any impact on our result, since in both cases the pressure was determined in-situ.

The pressure and temperature dependent XANES spectra were simulated by ab-initio code implemented in the FDMNES [33,34] package using multiple scattering theory.

## 3. Temperature dependent valence

The dependence of the Eu valence in $EuPt_2Si_2$ on the temperature-induced lattice contraction was determined using the element and orbital selectivity of X-ray absorption spectroscopy. Fig. 1 shows the Eu–$L_3$ XANES spectra for $EuPt_2Si_2$ as a function of temperature. The contribution from $Eu^{2+}$ ($5d^04f^7$) and $Eu^{3+}$ ($5d^14f^6$) can be easily distinguished since their threshold energies are separated by 8.5 eV due to the different Coulomb shielding in the X-ray excited state. Furthermore, we observe that the intensity of these two peaks display a temperature dependence, specially below 100 K. As in this material there is only one crystallographic site for Eu ions, this result is a clear indication that Eu presents a valence instability. While experimental XANES data of reference compounds with known valence state, such as $Eu_2O_3$, can be used to determine the degree of mixed valency by a linear combination of spectra, an accurate estimate must also consider the effects of crystal structure upon the XANES spectra [11,27]. To overcome this limitation, we have performed first principle calculations of the XANES spectra for both possible Eu valence configurations (2+ and 3+) using the lattice parameters determined by XRD for each temperature. Then, both theoretical $Eu^{2+}$ and $Eu^{3+}$ XANES spectra were linearly combined to reconstruct the experimental data, see Fig. 1(b). This method takes into account the different shape of XANES spectra corresponding to the two valences, which is ignored in the method of fitting the experimental data with Gaussian peaks [11] The behavior of the calculated valence is presented in Fig. 1(c), where we clearly observe an increase of Europium valence (from 2.27 at 300 K to 2.41 at 6 K) as a function of lattice contraction induced by the temperature decrease. Since this change on the valence is partial and continuous with the temperature decrease, it should not induce any abrupt change on the macroscopic properties of the material, in agreement with previous reports that did not observe any anomaly on magnetization, specific
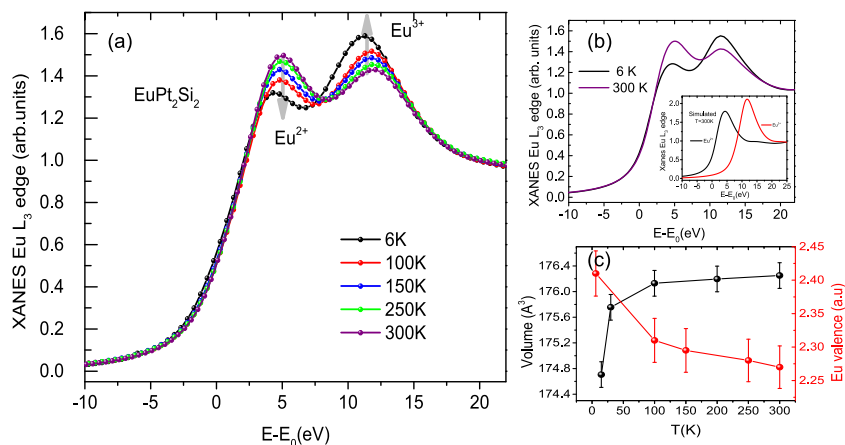
**Fig. 1.** (a) Experimental XANES Europium $L_3$ edge as function of temperature for EuPt$_2$Si$_2$ compound. (b) Linear combination of the simulated XANES spectra for two representative temperatures. The insert shows the simulated spectra for the Eu$^{2+}$ and Eu$^{3+}$ valences at 300 K (c) The behavior of Eu valence and the sample volume as a function of temperature for the compound.

heat or resistivity measurement [17,24]. Also, the fact that above 100 K the average Europium valence is almost constant, close to the divalent state, explains why the magnetization curves follow the Curie Weiss with the effective moment close to the Eu$^{2+}$ state. Our valence determination for high temperatures (T < 100 K) agrees well with the value reported by Takeuchi et al. [19]. On the other hand, previous work on a polycrystalline sample of EuPt$_2$Si$_2$ [23] reported a stable divalent state for this compound. One possible scenario for explaining the differences is that in both, our work and Takeuchi [19], single crystal samples were studied while in the Nagarajan's work sample were polycrystalline. The different samples can have slightly distinct lattice parameters and as we will discuss later in this manuscript this has a direct relation with the Europium valence on the EuPt$_2$Si$_2$ compound. On the other hand, for low temperature, while in the Takeuchi works the Europium valence was estimated to be 2.26 at 25 K, our results show that at 6 K the Europium valence is about 2.41. This apparent discrepancies in the low temperature valence determination can be explained by the different temperatures in which the two measurements were collected. As shown in 1(c) the volume suffers a more pronounced reduction below 50 K, and consequently a more significant effect on the Europium valence. We note that while the possible presence of Eu$_2$O$_3$ in the surface of the sample may influence on the observed value of Europium valence at ambient temperature and pressure conditions, this would not affect the increase of the Europium valence with decrease of temperature observed here. These results at ambient pressure revealed that the valence states of Europium are directly related to the lattice parameters, indicating an electronic instability takes place to accommodate the temperature dependent lattice contraction.

## 4. Pressure dependent valence

Owing to this electronic structure instability, the method of applying external pressure appears as a promising way to tune the lattice parameters and the corresponding electronic states to investigate if new ground states can be induced in this material. In this sense, firstly we determined the behavior of the crystal structure under pressure by means of in-situ high pressure synchrotron X-ray diffraction at low temperatures (40 K and 7 K) under pressures up to 27 GPa. The X-ray diffractograms (not shown here) preserve all the peaks in the entire range of pressure studied, indicating that no structural phase transition takes place up to the highest pressure. The pressure-dependent unit cell volume is presented in Fig. 2(c). It is noteworthy, that the unit cell volume is decreased by about 10% at highest pressure, which is much more dramatic than the contraction driven by temperature (<1%) and, therefore it is expected to induce more pronounced changes to the electronic structure in the material.

The electronic structure as a function of pressure was probed using the element and orbital selectivity of XAS technique. The XANES results for Eu–$L_3$ edge at 5 K are shown on Fig. 2(a). Our pressure dependent results show similar behavior to the XANES data as a function of temperature (Fig. 1). Although with more dramatic changes in the spectral weight transfer between Eu$^{2+}$ and Eu$^{3+}$ valences as pressure is increased, as expected due to the more pronounced contraction of unit cell relative to the temperature induced lattice contraction. The europium valence, determined in the same way as described in Section 3, abruptly changes from 2.4 at ambient pressure to 2.7 at 5 GPa, and linearly increases until becomes almost saturated (3+) at 27 GPa. After reaching about 27 GPa, the pressure was released and the effects on europium valence confirm to be reversible (red curve).

The spin-dependent sensitivity and atomic selectivity of XMCD were used to probe the magnetic properties of Eu and Pt ions, separately. In a similar fashion to the spin-averaged XANES, XMCD spectra probe the spin-dependent density of states (DOS) near the Fermi level. Fig. 2(b) presents the XMCD spectra at Eu $L_3$-edge as a function of applied pressure. The Eu$^{3+}$ contribution (due to its $4f^6$ weak Van Vleck paramagnetism) to the XMCD signal is mostly negligible when compared to the Eu$^{2+}$ strong local moment [11,26]. The latter is drastically affected by pressure as the valence changes against compression (Fig. 2(d)), indicating that the collapse of the ordered magnetic moment is due to a reduction of the fraction of magnetic ions (Eu$^{2+}$) induced by the lattice contraction. Such increase of valence has been observed in many other Eu-based valence fluctuating compounds [20,27,35,36].

In view of these Eu site results, one would expect some changes in the hybridization between Eu and Pt orbitals and consequently a modification of the electronic structure of Pt states, in a similar scenario to what was observed for Ga polarized states in Eu$_{0.5}$Yb$_{0.5}$Ga$_4$ [25]. In this context, we performed pressure and temperature dependent XANES experiments at Pt $L_{2,3}$ edges as well as XMCD experiments at ambient pressure and low temperature, as shown in Fig. 3. In the XANES spectra no significant change is observed as a function of either temperature or pressure, indicating that the changes in Europium valence do not induce any pronounced modifications in the electronic configuration of the platinum ion. As per the magnetic properties of Pt are concerned, the observed Pt $L_{2,3}$-edges XMCD signal, shown in Fig. 3, is quite small. Using the XMCD sum rules [37,38] we can estimate that the total Pt moment at 4 T is around 0.02 $\mu_B$, which is 60 times lower than the magnetic moment of the sample at this magnetic field (1.2 $\mu_B$) obtained from the magnetization experiments. Based on these XANES and XMCD data we can conclude that the observed valence change in Eu induced by both temperature and pressure does not affect the electronic configuration of Pt ions and that Pt has negligible contribution to the magnetic properties of this material.
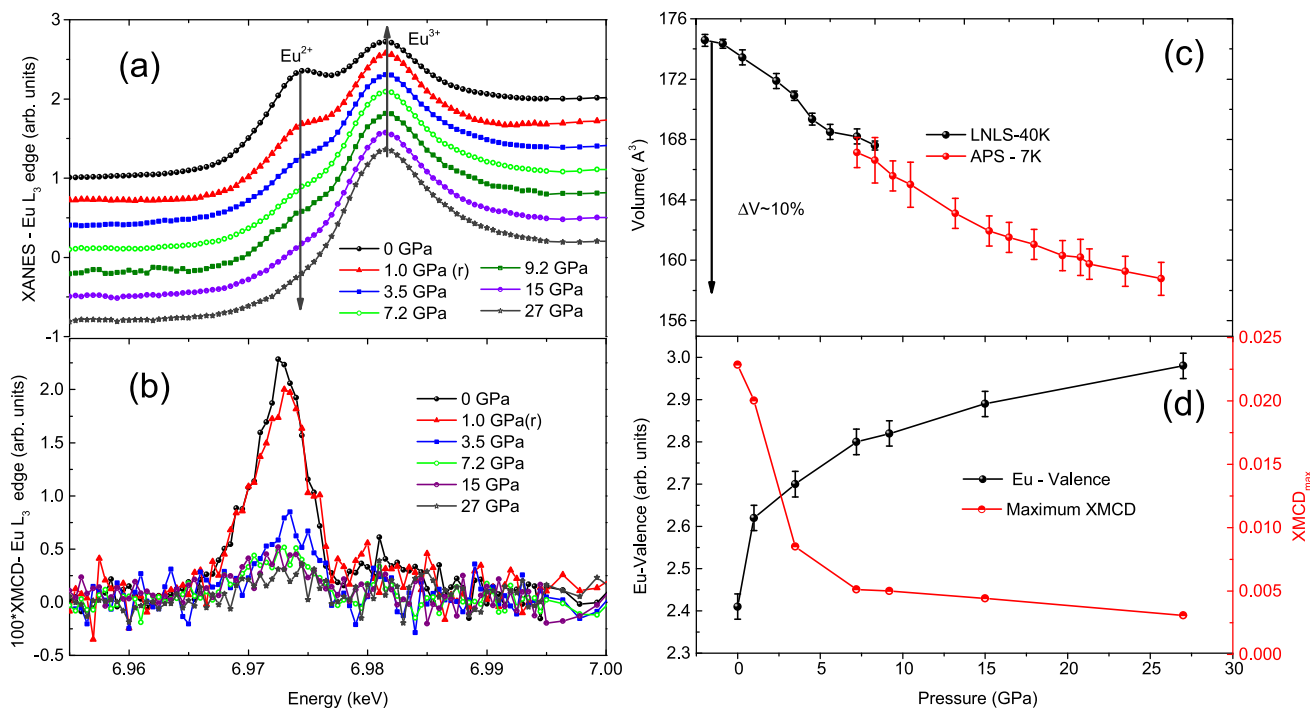
**Fig. 2.** (a) Europium $L_3$ XANES spectra as a function of pressure on $EuPt_2Si_2$ sample at 5 K. The lattice contraction induced by pressure results in a suppression of the peak on the Europium XANES spectra related to the $Eu^{2+}$ valence. (b) Pressure dependence of XMCD (X-ray Magnetic Circular Dichroism) data, taken at the Eu $L_3$ absorption edge ($2p_{3/2}$ initial state) of the $EuPt_2Si_2$ compound at 5 K. It is clear that initially the XMCD signal sharply decreases with pressure until 7.2 GPa and then remains constant up to 27 GPa. The reduction of XMCD signal is related to the Europium valence change from 2+ (magnetic) to 3+ (non magnetic). The observed effects in both XANES and XMCD data are reversible as evidenced by the 1 GPa point after pressure release. (c) The pressure–volume dependence up to 31 GPa. (d) Behavior of the valence and the maximum XMCD signal as a function of pressure. In all figures solid lines are guide for the eye.

## 5. Discussion

The results presented here demonstrate the instability of the Europium valence in the $EuPt_2Si_2$ compound either by variation of temperature or application of pressure. In contrast with other Europium based compounds that present a first order valence transition [15,16,19,20,39], here the Europium valence is directly connected to the unit cell volume, in such way that when the cell is contracted, either by temperature or pressure, the valence is increased. The precise correspondence between the Europium valence and the sample volume is illustrated in Fig. 4, which was estimated from our XRD and XAS data. The continuous pressure induced valence change has a direct correlation with the change on the magnetic properties. The Eu valence change from 2+ to 3+ reduces the fraction of magnetic Eu ions in the material which leads to a suppression of the magnetic response. This scenario is different to the observed for the other compounds of the same structure, such as $EuRh_2Si_2$ and $EuNi_2Ge_2$ [21], where the pressure first induces a suppression of the magnetic ordering (at 0.8 GPa for $EuRh_2Si_2$ and at 2 GPa for $EuNi_2Ge_2$) and then at the higher pressures a first order valence transition (from 2+ to 3+) occurs. On top of that we might speculate that the pressure induced valence change on the Europium ion from 2+ to 3+ is also responsible for the previous observation of the expressive reduction of the resistivity of the compound at low temperatures, since the application of pressure leads to the electrons initially at Eu-4$f$ orbital being increasingly transferred to the conduction band which entails an increase in the number of charge carriers on the material.

## 6. Summary

In summary we used the element- and orbital- selectivity of XANES and XMCD measurements on Eu and Pt $L_3$ absorption edges to probe the electronic and magnetic properties of the $EuPt_2Si_2$ compound under lattice contraction induced by temperature and pressure. We demonstrate

that this compound has an unstable valence which is directly related to the unit cell lattice parameters. For both temperature decrease and pressure increase we observed a continuous valence change from $2^+$ to $3^+$. In addition, we can argue that the reduction of the residual resistivity ratio as a function of pressure previously reported for this material [17,24], is due to the increase of charge carriers on the material because the Eu-4$f$ charges are transferred to the conduction band. This complete spectroscopic study of the electronic structure and magnetic properties under high pressure should guide efforts in understanding valence instabilities and related changes in exchange interactions in similar rare earth intermetallic systems when undergo a contraction in unit-cell volume.
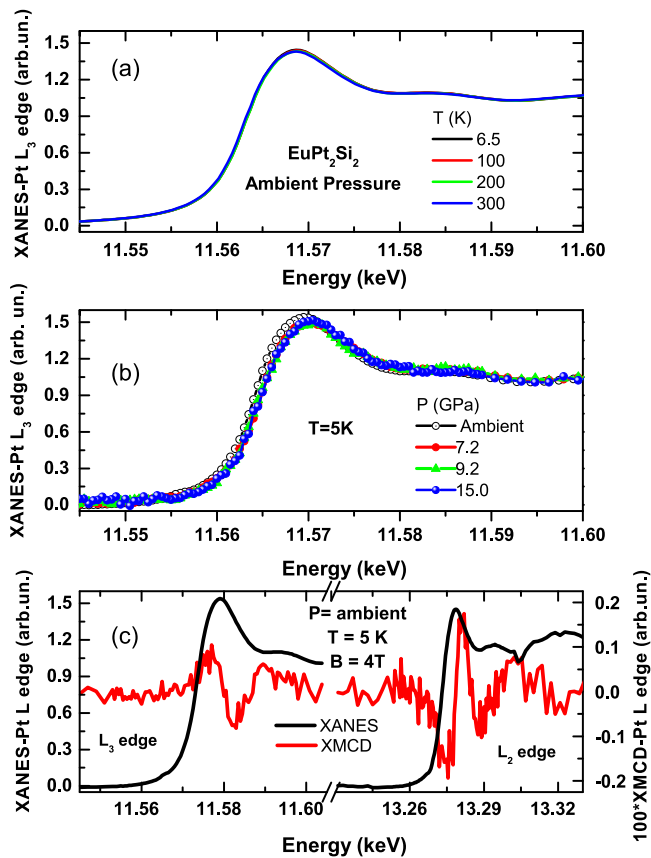
**Fig. 3.** (a) Platinum $L_3$ XANES spectra as a function of temperature. (b) Pressure dependence of Platinum $L_3$ XANES spectra. (c) Platinum $L_3$ and $L_2$ XMCD spectra at ambient pressure.
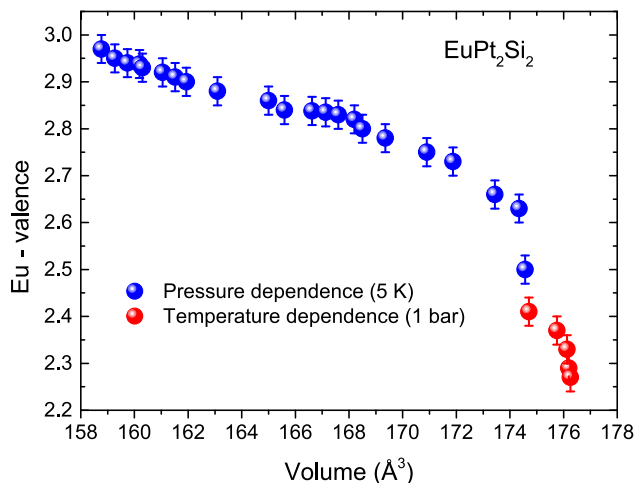


**Fig. 4.** Europium valence as a function of volume for the $EuPt_2Si_2$ compound.

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