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# Raman Effect During Formation of the Ordered $L1_0$-FePd Phase

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Effect of the hydrogen heat treatment compared with vacuum annealing in the temperature range of 500–700°C on the formation of the ordered $L1_0$-FePd phase and variations in Raman spectra of the equiatomic FePd films are studied. The hydrogen atoms introduced into the nanoscale FePd films change the electronic structure and magnetic properties and states of the film. Ordering processes are accelerated under hydrogen treatments compared with vacuum annealing. By changing of parameters of the hydrogen annealing of the FePd film, it is possible to control the phase composition, ferromagnet ↔ paramagnet-variation in the magnetic states. The Raman spectroscopy allows investigate the dynamics of structural changes in the FePd films during the ordering and the ordered $L1_0$-FePd-phase formation.

Досліджено вплив водневого термічного оброблення порівняно з відпалом у вакуумі в інтервалі температур 500–700°C на формування впорядкованої фази $L1_0$-FePd та зміну спектрів комбінаційного розсіяння світла в еквіатомних плівках FePd. Атоми Гідроґену, введені в нанорозмірні плівки FePd, змінили електронну структуру, магнетні властивості та стани плівки. Процеси впорядкування пришвидшуються під час оброблення воднем порівняно з вакуумним відпалом. Змінюючи параметри відпалу плівки FePd у водні, можна керувати фазовим складом, зміною феромагнетик ↔ парамагнетик магнетних станів. Раманова спектроскопія уможливлює дослідити динаміку структурних змін плівок FePd під час упорядкування й утворення впорядкованої фази $L1_0$-FePd.

**Key words:** hydrogen, ordering, coercivity, Raman scattering spectroscopy, paramagnetism, hard-magnetic material, $L1_0$-FePd, magnetization.

**Ключові слова:** водень, впорядкування, коерцитивна сила, Раманова спектроскопія комбінаційного розсіяння, парамагнетик, магнетотвердий матеріял, $L1_0$-FePd, намагнетованість.

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## 1. INTRODUCTION

The ordered $L1_0$-FePd films are a promising material for use as magnetic media with ultra-high density, and in spintronics devices [1–3]. Usually, the FePd films deposited at room temperature have a (111) texture and soft-magnetic properties [1, 4, 5]. The annealing environment significantly affects the structure, magnetic and optical properties of the films [4–8].

It was found that hydrogenation increases the coercivity and residual magnetization in films of alloys enriched with Pd [9]. Electron–phonon interactions in the magnetic equiatomic layered $L1_0$-FePd structure were studied in Ref. [10]. Position of H in the structure has a direct influence on the Curie temperature $T_C$ values and the suppression of magnetism. Annealing-induced cyclic enhancement of the coercivity is related to the competition between thermal activation and H binding [11]. Magnetic films based on Pd can provide the ability to manipulate magnetic states using a current of spin–orbit torques [12]. The authors of Ref. [13] theoretically and experimentally investigated the disappearance of magnetism in Fe–H at a pressure of approximately 28 GPa and revealed the existence of a magnetic transition from a ferromagnetic state to a paramagnetic one, which was caused by pressure. According to the calculations, the H absorption is a favourable process in both the $A1$-FePd phase and $L1_0$-FePd one [13]. The authors showed that the location of hydrogen atom in the tetrahedral interstitial site of the $A1$ phase is more stable than in the octahedral interstitial site in the $L1_0$-phase lattice. The influence of hydrogen on magnetism is reversible [15–17].

In Ref. [16], a hydrogen-sensitive FePd alloy film was deposited on a Co/[Pt/Co]$_4$/Pt multilayer substrate with perpendicular magnetic anisotropy. As a result of hydrogenation, a spin reorientation transition from the perpendicular to the planar direction in the 2-nm thick FePd layer was observed. These findings are valuable for applications in spintronics using electronic control of hydrogen atom migration.

For obtaining the desired properties and multifunctionality, it is necessary to understand the interaction between the material structure and functionality.

Surface-enhanced Raman spectroscopy (SERS) is a powerful analytical method that provides amplification of the Raman signal by an order of $10^6$–$10^{14}$ [18]. New nanostructured materials are being developed for use as highly sensitive substrates for SERS [18]. An innovative substrate based on a nanoporous FePd alloy, which enhances surface Raman scattering, has ferromagnetic properties and can be used as a quantitative tool.

The aim of this work is to study the effect of hydrogen treatment compare the vacuum annealing on the ordered $L1_0$-FePd phase formation and application of the Raman scattering for investigation of dynamic of ordering processes.

## 2. EXPERIMENTAL

Equiatomic nanoscale FePd films by thickness of 5 nm were deposited at room temperature on the $SiO_2$/Si(001) substrates by magnetron co-sputtering from separate Fe and Pd targets. The film was deposited under an Ar working pressure of $3.5 \cdot 10^{-1}$ Pa in a chamber with a starting ultra-high vacuum of $3 \cdot 10^{-6}$ Pa. The deposition rate and layer thicknesses were monitored using a quartz resonator. In addition, the composition of the deposited films was controlled using Rutherford backscattering spectrometry (RBS). After deposition, the samples were annealed in vacuum ($P = 10^{-3}$ Pa) and in hydrogen (pressure $\cong 1$ atm) for 1–2 h in a temperature range of 500–700°C.

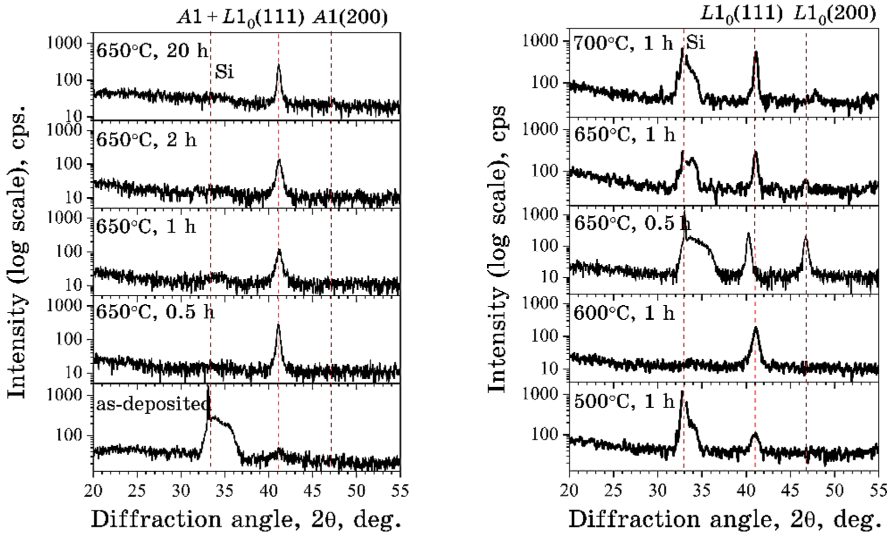
The phase composition and structure of the films after deposition and annealing were studied by the x-ray diffraction on ULTIMA IV (Rigaku) diffractometer (Cu$K_\alpha$ radiation). Magnetic measurements were performed using a vibrating sample magnetometer (SQUID-VSM). All measurements were performed at room temperature.

Structural changes in the FePd films was investigated by Raman spectroscopy at a Renishaw InVia Raman Microscope spectrometer with the optical excitation wavelength of 785 nm and an analysis of the frequency criterion of radiation scattering [20].

## 3. RESULTS AND DISCUSSION

### 3.1. Processes of Ordering in FePd Films during Annealing in Vacuum

Figure 1, *a* shows the XRD patterns of the as-deposited FePd films and annealed in vacuum at 650°C for 0.5–20 h. After deposition, the weak FePd(111) peak and reflection from the substrate are observed (Fig. 1, *a*). The coercivity of the soft-magnetic FePd film is of 177 Oe (Fig. 2, *a*). The intensity of FePd(111) peak increases af-



*a* *b*

**Fig. 1.** XRD patterns of the as-deposited FePd (5 nm) films, after annealing in vacuum at 650°C for 0.5–20 h (*a*) and in hydrogen at 500–700°C for 0.5–1 h (*b*), Cu$K_\alpha$ radiation.

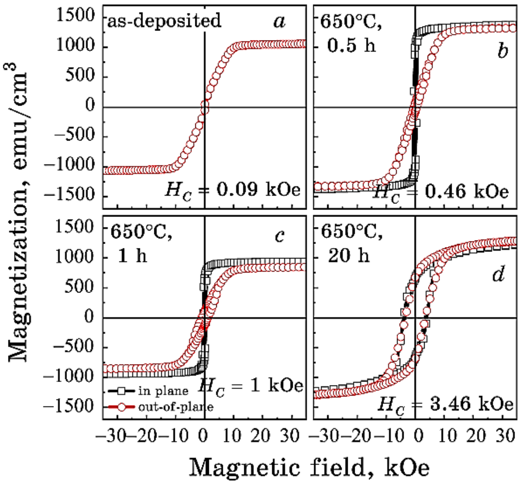


**Fig. 2.** $M$–$H$-hysteresis loops of FePd films after deposition (*a*) and annealing in vacuum at 650°C for 0.5 h (*b*), 1 h (*c*) and 20 h (*d*).

ter annealing at 650°C for 0.5–20 h that indicates preferential grains growth with (111) texture. After annealing, this reflection can be belonging to either disordered $A1$- or the ordered $L1_0$-FePd phases.

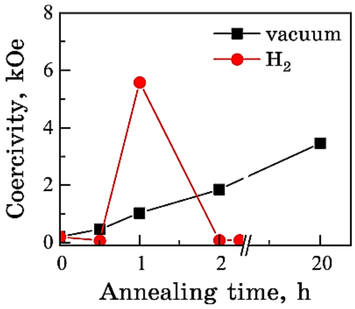


**Fig. 3.** Changes in coercivity value of the FePd films on annealing time in vacuum and hydrogen at 650°C.

However, regardless of the absence of the superstructural FePd(001) and fundamental FePd(002) peaks, the hard magnetic $L1_0$-FePd phase is formed according to the measurements of magnetic properties (Fig. 2, *b*, *c*, *d*). The film after annealing in vacuum for 20 h is magnetically isotropic (Fig. 2, *d*).

Figure 3 shows the change in the coercivity of FePd films annealed in vacuum and hydrogen atmospheres in dependence on the time of annealing. The coercivity of the film after annealing in vacuum at 650°C for 1 h is of about 1 kOe that indicates the hard-magnetic $L1_0$-phase formation (Fig. 2, *c*, Fig. 3). Volume fraction of this phase increases with an increase in the annealing time to 20 h, and the coercivity reaches 3.46 kOe (Fig. 2, *d*, Fig. 3).

### 3.2. Processes of Ordering in FePd Films during Annealing in Hydrogen

Processes of ordering in FePd films during annealing in hydrogen differ from annealing in vacuum. The intensity of the (111) reflection increased after heat treatment in the temperature range of 500–600°C for 1 h (Fig. 1, *b*). The grains of the $L1_0$-FePd phase have a (111) texture. After annealing at 600°C, the $L1_0$ phase is formed and the coercivity becomes of 0.9 kOe (Fig. 4, *a*).

The (111) peak shifts toward smaller angles and (200) peak appears after annealing at 650°C for 0.5 h (Fig. 1, *b*). This indicates an increase in the lattice parameters and unit cell volume of the $A1$-FePd phase. However, the magnetic properties become uncharacteristic for both the $A1$- and $L1_0$-FePd phases (Fig. 4, *b*).

A hard-magnetic $L1_0$-FePd phase formed after annealing at 650°C for 1 h has a coercivity value of 5.5 kOe (Fig. 1, *b*, Fig. 4, *c*). Increasing the annealing time to 2 h resulted in the film becoming x-ray amorphous and losing its hard-magnetic properties (Fig. 4, *d*).

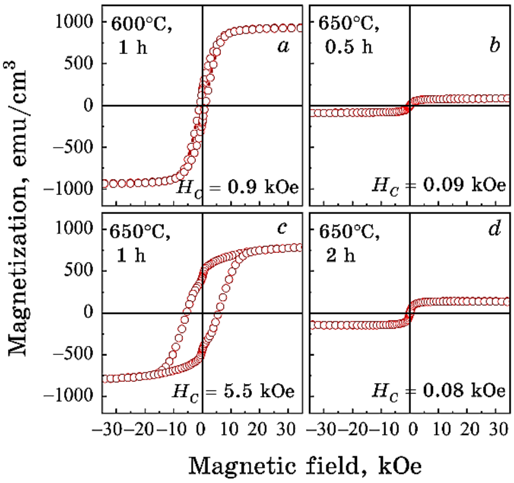


**Fig. 4.** $M$–$H$-hysteresis loops of FePd films annealed in hydrogen at 600°C for 1 h (*a*), at 650°C for 0.5 h (*b*), 1 h (*c*) and 2 h (*d*).

The magnetic state of the FePd film changed from hard-magnetic state to paramagnetic one as a result of incorporation of hydrogen atoms into the film. A further increase in the annealing temperature to 700°C led to rise in the (111) peak intensity.

### 3.3. Raman Scattering in FePd Films after Annealing in Vacuum and Hydrogen

Figure 5, *a* shows the Raman spectra of as-deposited sample irradiated from two sides, namely, from the substrate and from the film and annealed in vacuum at 650°C. The narrow spectrum at a frequency of 530 cm$^{-1}$ corresponds to silicon. The next two resonance peaks of the spectrum can be attributed to oxides and the last peak to the luminescence of the substrate, which is caused by defects (inhomogeneities) in the silicon structure. The hydrogen atoms introduced into the FePd phase change the electronic structure and magnetic properties of the film [14]. After annealing in vacuum at 650°C, the magnetic properties correspond to a two-phase state in the film (Fig. 2, *b*, *c*). The luminescence peak is structurally sensitive to external influences. Its amplitude is greater than for pure substrate. This effect is caused by the enhancement of luminescence due to the presence of the film. The frequency shift of optical spectra indicates the appearance of stimulated Raman scattering, which enhances the oscillations of atoms or molecules during the ordering process. Figure 5, *b* shows the results of Raman spectroscopy for the FePd films annealed at different temperatures in hydrogen.

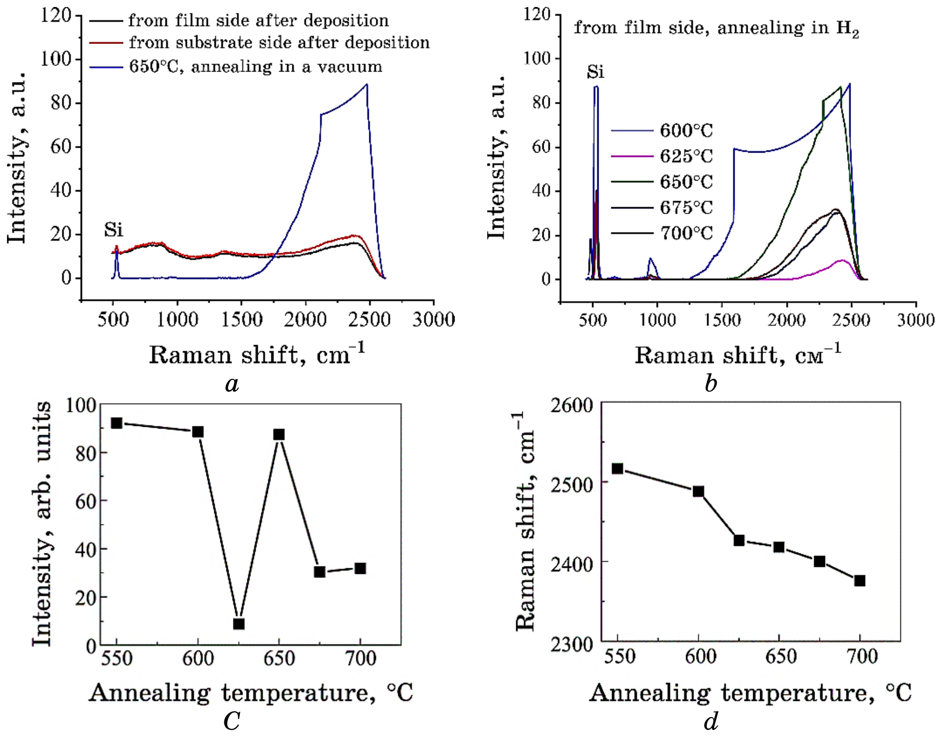


**Fig. 5.** Raman spectra of magnetic FePd film after deposition and annealing in vacuum at 650°C (*a*), after annealing in hydrogen at 600–700°C for 1 h (*b*); dependences of the intensity (*c*) and position (Raman shift) of the resonance peak (*d*) on annealing temperature [17].

There are maxima at $\cong 2376$–$2516$ cm$^{-1}$, which indicate the excitation of coupled $E$ modes of Pd and Fe oscillations. There is a broad peak of significant intensity in the range 1594–2488 cm$^{-1}$ of the spectrum of the FePd film annealed in hydrogen at 600°C (Fig. 5, *b*).

Such a broadening can occur because of the diffusion of hydrogen atoms into the film and the beginning of the ordered $L1_0$-FePd phase. The resonant frequency peak from the film has an asymmetric shape in the frequency range 1500–2600 cm$^{-1}$. The asymmetry of the Raman spectrum is related to the two-phase state ($A1 + L1_0$) that is present in the film. Annealing in hydrogen atmosphere led to the disappearance of an oxide peaks that can be explained by the reduction properties of hydrogen (Fig. 5, *b*). The oxidation process was suppressed by annealing in an $Ar + H_2$ (3 vol.%) atmosphere [11]. With increasing temperature of the ordering process, the number of grains of the ordered phase increases and the width of the resonance Raman peak decreases (Fig. 5, *b*). The width of the resonance peak and its variations in the temperature range 500–700°C are associated with the ordering process and formation of the hard magnetic $L1_0$ phase from the disordered $A1$-FePd phase during annealing of the FePd films both in vacuum (Fig. 5, *a*) and in hydrogen (Fig. 5, *b*).

The results show that during annealing in hydrogen atmosphere, the position of the peak at 2376–2516 $\mathrm{cm}^{-1}$ corresponds to the formation of the ordered $L1_0$-FePd phase, and its width decreases with increasing annealing temperature (Fig. 5, *b*). The resonance peak at $\cong 2500$ $\mathrm{cm}^{-1}$ corresponds to the ordered $L1_0$-FePd phase, and the less intense broad part of the peak corresponds to the disordered $A1$-FePd phase in the zone with a different ordering degree. As the amount of ordered phase increases, this peak shifts to higher frequencies (Fig. 5, *b*). After annealing in hydrogen atmosphere at 625°C, there is a sharp decrease in the intensity of the peaks, which is due to the influence of the hydrogen atoms on the electronic structure of the FePd film (Fig. 5, *b*, *c*). An increase in the intensity of oscillations at $\cong 2418$ $\mathrm{cm}^{-1}$ and a decrease in the width of this peak after annealing in hydrogen at 650°C, compared to annealing in vacuum, indicates increased amount of the ordered $L1_0$ phase in the FePd film (Fig. 5, *a*–*c*). The luminescence peak with higher intensity corresponds to the state of the film with the largest amount of the $L1_0$ phase both after annealing in vacuum and in hydrogen at 650°C for 1 h (Fig. 5, *b*, *c*). In this case, the resonance peak from the film remains slightly asymmetric, which is caused by the residual amount of the $A1$-FePd phase. After annealing at 675°C and 700°C, there is a broad peak of lower intensity in the range 2376–2400 $\mathrm{cm}^{-1}$, which may correspond to the oscillations of excess iron atoms. The changes in the intensity of the Raman scattering spectra of the FePd film with the annealing temperature correspond to the changes in the magnetic states in the film (Fig. 5, *c*).

However, the intensity of the Raman resonance peak changes nonlinearly with an increase in the temperature of the heat treatment in hydrogen atmosphere (Fig. 5, *c*). The ferromagnetic state in a film with uncompensated spins of Fe atoms leads to an increased intensity of the resonance signal of the Raman spectrum compared to the paramagnetic state in which the polarization is insignificant. The intensity minima in the Raman spectra at 625, 675 and 700°C correspond to paramagnetic states. Figure 5, *d* shows the changes in the resonance peak position on the Raman spectra of the magnetic FePd film after annealing in hydrogen atmosphere on annealing temperature. As the annealing temperature increases, the value of the Raman shift reduces due to an increase in the amount of ordered $L1_0$-FePd phase. The Raman shift changes almost linearly with the annealing temperature and does not reflect changes in the magnetic states of the film (Fig. 5, *d*).

The obtained results show that hydrogen atoms incorporated in the FePd film during annealing in the temperature range of 500–700°C have a significant effect not only on the dynamics of ordering processes, structure, but also on the reversibility of the ferromagnetic ↔ paramagnetic state compared to annealing in vacuum [16]. The ordering process is influenced by two factors, namely, the effect of introduced hydrogen atoms on the electronic structure of the film and the change in the volume of the unit cell during the $A1$-FePd-to-$L1_0$-FePd transformation.

In the as-deposited FePd film, a disordered soft-magnetic $A1$-FePd phase forms. There are five electrons with positive spins and one with negative spin on the 3*d* shell in the outer layer of iron atom; as a result, four spins remain uncompensated. The exchange energy between two neighbouring Fe atoms causes atom polarization and leads to a ferromagnetic state. Annealing in hydrogen atmosphere is accompanied by the incorporation of hydrogen atoms into the film. Hydrogen atoms are located in octahedral and tetrahedral interstitial sites and increase the volume of the $A1$-FePd phase unit cell. This weakens atomic bonds and accelerates the formation of the $L1_0$-FePd phase. The soft-magnetic state in the film preserves up to annealing at 600°C for 1 h (Fig. 4, *a*). Hydrogen atom introduced in the film gives its electron to Fe atom. As a result, the film electronic structure changes. When the hydrogen concentration in the film changes with increase in annealing time or temperature and the process of ordering and formation of $L1_0$ grains develops, the magnetic properties and states change: soft-magnetic $A1$ (as-deposited film) → soft-magnetic $A1$ + hard-magnetic $L1_0$ (600°C, 1 h) → paramagnetic (625°C, 1 h–650°C, 0.5 h) → hard-magnetic $L1_0$ (650°C, 1 h) → paramagnetic (650°C, 2 h). With the gradual filling of the inner *d*-shell of Fe atoms, part of the positive uncompensated spins becomes compensated ones. After annealing at a temperature of 625°C for 1 h or at 650°C for 0.5 h, the magnetic state of the film changes to paramagnetic. As the annealing time at 650°C increases to 1 h, the volume of the lattice cell decreases as the $A1 \rightarrow L1_0$-ordering process develops, and the second necessary factor $a/r$ (the ratio of the interatomic distance to the radius of the unfilled shell of Fe atoms) for the appearance of ferromagnetism begins to act. A hard magnetic state appears in the film. Further filling of the 3*d* shell of Fe atoms with hydrogen electrons after annealing at 700°C for 1 h is accompanied by the reappearance of the paramagnetic state. During annealing in vacuum, the ordering process ($A1 \rightarrow L1_0$) proceeds more slowly than in hydrogen, and the change from the soft-magnetic state to the hard-magnetic state is affected only by the decrease in the volume of the unit cell during the formation of the $L1_0$-FePd phase.

The highest values of the coercivity in the film at $\cong 5.5$ kOe are observed after annealing in hydrogen at 650°C for 1 h, which is 5-times higher in comparison with annealing in vacuum. The ferromagnetic state in the film with uncompensated spins of Fe atoms leads to an increase in the intensity of the resonance spectrum of the Raman signal compared to the paramagnetic state in which the polarization is insignificant. The shift in the frequency of scattered light during the $A1 \rightarrow L1_0$-FePd solid-state transformation is a manifestation of forced scattering of optical radiation. It is advisable to use the SERS method to study the dynamics of structural changes in magnetic FePd films during the ordering process. By changing the annealing parameters of the equiatomic FePd film in hydrogen, it is possible to control the phase composition, magnetic properties and states. The use of the FePd film as a template enhances surface Raman scattering and increases the sensitivity for organic objects. Reversible ferromagnet ↔ paramagnet change of the film magnetic state can find practical application in nanoelectronics.

## 4. CONCLUSION

Annealing atmosphere, temperature and time of annealing affect the ordered $L1_0$-phase formation, structure, magnetic properties and states of the equiatomic FePd films. As revealed, the annealing in hydrogen at 500–700°C results in the reversible change of the magnetic state of the FePd films: soft-magnetic → soft-magnetic + hard-magnetic → paramagnetic → hard-magnetic → paramagnetic. Obviously, this is caused by changes in electronic structure.

The SERS is promising method for studying of the ordering process and dynamics of the structural and magnetic changes in the FePd films. Use of the FePd films as templates enhances the surface Raman scattering and sensitivity for organic objects.

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