

# Characterization of Fe/W Spin-Polarized Tips by Means of Holographic TEM and Spin-Polarized STS of Optically Pumped p-GaAs

T. Matsuda<sup>1</sup>, A. Tonomura<sup>2</sup>, T. K. Yamada<sup>3</sup>, D. Okuyama<sup>3</sup>, N. Mizuno<sup>3</sup>, A. L. Vazquez V. de Parga<sup>4</sup>, H. van Kempen<sup>5</sup>, and T. Mizoguchi<sup>3</sup>

<sup>1</sup>Frontier Research System, The Institute of Physical and Chemical Research, Hatoyama, Saitama 350-0395, Japan, and Naka Customer Center, Hitachi Science Systems, Ltd., Ishikawa, Hitachinaka, Ibaraki 312-0057, Japan

<sup>2</sup>Advanced Research Laboratory, Hitachi Ltd., Hatoyama, Saitama 350-0395, Japan

<sup>3</sup>Faculty of Science, Gakushuin University, 1-5-1 Mejiro, 171-8588 Tokyo, Japan

<sup>4</sup>Departamento Fisica de la Materia Condensada, Universidad Autonoma de Madrid, Madrid E-28049, Spain

<sup>5</sup>Institute for Molecules and Materials, Radboud University, 6525 ED Nijmegen, The Netherlands

**Holographic transmission electron microscopy showed that magnetic flux does not diverge from the top of an Fe-coated W tip indicating the in-plane magnetization at the apex of successful spin-polarized tips. A dependence of differential conductivities of GaAs with Fe-coated W tips on the helicity of illuminating laser light was confirmed at negative sample bias where partially spin-polarized electrons in the conduction band of p-GaAs tunnel into spin-dependent Fe empty states while no dependence of those with nonmagnetic W tips.**

**Index Terms**—Magnetic field measurement, optical pumping, spectroscopy, tunneling.

## I. INTRODUCTION

**A**MONG remarkable progress in wide application of scanning probe microscopy, spin polarized scanning tunneling microscopy and spectroscopy (SP-STM/STS) is now of great interest as it gives us the local spin density of states with atomic scale resolution [1]–[3].

There is a limited number of groups that have established the SP-STM/STS technique with which they succeed to get magnetic images of samples, but the importance of the characterization of spin-polarized tips is not yet recognized satisfactorily. The asymmetry of magnetic contrast in SP-STM/STS is considered to be a product of the polarization of the sample surface and that of the apex of the tip and  $\cos\theta$ , where  $\theta$  is the angle between the quantization axes of the polarization of a sample and the apex of a tip. We have succeeded in obtaining images of magnetic contrast on antiferromagnetic Mn layer stackings on Fe(001) [2] with Fe-coated W tips which are widely used to get magnetic contrast on samples with in-plane magnetizations.

In order to investigate nanoscale local magnetization at the apex of the Fe/W tip, we observed directly the magnetic flux from the tip by means of electron-holographic interference microscopy.

On the other hand, after reports of spin-dependent tunneling of optically excited electrons for junctions [4], [5], extensive efforts [6]–[9] have been dedicated to develop a spin-polarized STM with a GaAs microprobe tip. The GaAs tips made by

cleavage in air seem to have a serious problem since even non-magnetic Au test samples showed helicity-dependent optical scattering in the STM-junction [9]. However, use of optically pumped spin-polarized electrons from GaAs is still attractive since its spin polarization is theoretically known. Thus, the spin polarization of Fe-coated W tips is investigated in this study using polarized electrons from optically pumped p-GaAs(110) as the standard sample.

## II. EXPERIMENTAL DETAILS

Electron holographic interference microscopy measurements were performed by transmission electron microscopy (TEM) with a 300-keV field emission electron source and a TEM with a 1-MeV cold emission electron source at a pressure of about  $10^{-6}$  Pa. For holographic TEM measurements blunt W tips coated with 10-nm Fe film were prepared in ultrahigh vacuum (UHV) in the same way as we prepared our SP-STS experiments [1], [2]. They were covered by 2-nm Au film to prevent oxidization. Phase amplified ( $\times 16$ ) interferogram images show magnetic force lines as black–white contrasts.

A clean p-GaAs(110) surface was obtained by cleavage of a p-GaAs(001)-wafer in UHV ( $2 \times 10^{-7}$  Pa) [10]. SP-STS experiments on this p-GaAs(110), illuminated by circularly polarized laser light ( $\sim 0.2$  mW/mm<sup>2</sup>,  $\lambda = 780$  nm) incident with an angle of  $75^\circ$  relative to the surface normal with Fe-coated (4–10 nm) W tips and nonmagnetic W tip were performed at room temperature in UHV in a current imaging tunneling spectroscopy mode. An  $I(V)$  curve obtained by averaging over about 1000 pixels in a clean terrace of p-GaAs(110) was numerically differentiated to get a differential conductivity ( $dI/dV$ ) curve.

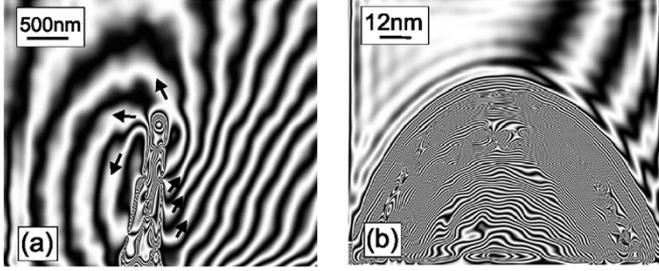


Fig. 1. Electron-holographic phase-amplified ( $\times 16$ ) interference microscopy results of an Fe-coated W tip. Black–white contrast corresponds to magnetic force lines. (a) was obtained by a TEM with a 300-keV field emission electron source. (b) was obtained by a TEM with a 1-MeV cold emission electron source. Single fringe corresponds to a magnetic flux of  $h/(16e)$ , where  $h$  denotes the Planck's constant and  $e$  the electron charge.

### III. HOLOGRAM TEM RESULTS

We might imagine that flux lines diverge from an end of a sharp ferromagnetic needle, but we should keep in mind that the SP-STM tip is not a bulk ferromagnet, but a very thin layer of ferromagnetic film, i.e., 10-nm Fe film on a nonmagnetic blunt tip of tungsten. This peculiar shape of ferromagnetic thin film, i.e., a thin pipe of radius about 130 nm terminated by a hemispherical cap, makes it possible to realize in-plane magnetization at the top of the apex. Holographic images of an Fe-coated W tip are shown in Fig. 1. Magnetic flux lines diverge from one side of the tip and go into the other side as seen in the overall view [Fig. 1(a)]. It is clear that the flux does not diverge at the top of apex of the tip as seen in the enlarged view [Fig. 1(b)], where the magnetization at the top of the tip is in-plane, i.e., perpendicular to the axis ( $z$  direction) of the tip. We measured four different Fe-coated W tips and obtained similar results.

If we take the direction of local magnetization at the top of apex of the tip as the  $x$  axis, the magnetization of one side ( $x < 0$ ) of thin Fe film on tungsten tip goes upward to  $z$  and goes downward on the other side ( $x > 0$ ) to the  $-z$  direction. Thus, there must be magnetic domain walls along  $z$  direction between them at  $x = 0$ . Since Fe film on the tungsten tip is thin enough, the domain walls must be Néel type with cross tie structure, from which the magnetic flux lines diverge or converge. The width of a Néel wall is expressed in a limiting case of thin films as [11]

$$\delta_N = \pi \sqrt{\frac{2A}{K}} \quad (\text{for film thickness} \ll \delta_N). \quad (1)$$

Inserting rough values close to those for  $\alpha$ -Fe,  $A \sim 1.5 \times 10^{-11}$  J/m and  $K \sim 4.7 \times 10^4$  J/m<sup>3</sup>, we get  $\delta_N \sim 80$  nm, which is about a one-tenth part of a circle of the Fe thin film on the top part of the tungsten tip. It would be interesting to investigate the micromagnetic structure of this peculiar shape of ferromagnetic thin sheath as a function of the curvature of the apex and thickness of the film.

Our approach is rather practical. We made successful Fe/W tips in SP-STM and investigate the micromagnetic structure by means of holographic TEM. This result explains why we cannot get good SP-STM results with sharp tips as used in conventional STM because the magnetization is undefined at an apex of a tip.

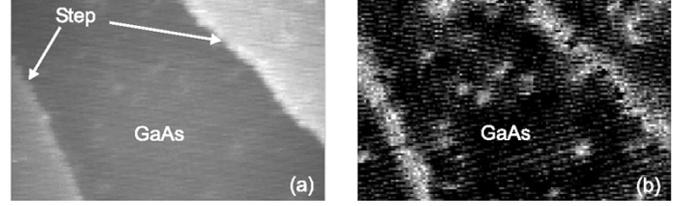


Fig. 2. (a) shows an STM image ( $54 \times 24$  nm<sup>2</sup>,  $V_s = +2.0$  V,  $I = 0.06$  nA) obtained on p-GaAs(110). (b) shows an  $I(V)$  map at  $-1$  V obtained on the same area as (b).

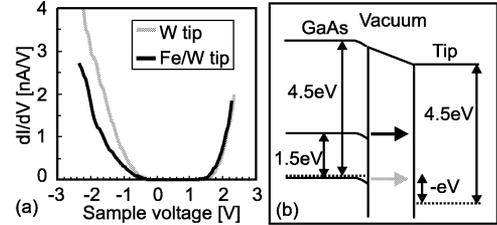


Fig. 3. (a)  $dI/dV$  curves obtained on GaAs(110) with a W tip (gray) and an Fe-coated W tip (black) without illumination of light. (b) Scheme of tunneling with illumination of circular polarized light at a negative sample bias voltage. Two channels of tunneling electrons are considered: electrons from the valence band (gray arrow) in addition to electrons from the conduction band (black arrow).

### IV. SPIN-POLARIZED STM/STS TIPS STUDIED BY SPIN-POLARIZED ELECTRONS FROM OPTICALLY PUMPED p-GaAs(110)

A topographic image of p-GaAs(110) cleaved in UHV [Fig. 2(a)] shows flat terraces of atomic planes. A spectroscopic image obtained at the same area [Fig. 2(b)] shows, however, several sites, not only steps, which look different from a pure atomic array of GaAs(110). We carefully select a pure clean area of an atomic plane for further spectroscopic study.

We are interested in electron tunneling from a p-GaAs sample to the W or Fe/W tips at negative sample bias voltage for characterizations of the tips. Without light illumination, electrons tunnel from valence states of p-GaAs to empty states of a tip at negative sample bias. Averaged  $dI/dV$  curves obtained with W or Fe/W tips from the clean GaAs surface without illumination of light are shown in Fig. 3(a). A rapid increase of  $dI/dV$  below the Fermi level and a bandgap of about 1.5 V above the Fermi energy are observed. The difference between W and Fe/W tips is likely caused by different work functions of W and Fe. Similar difference in  $dI/dV$  curves were observed on Mn(001) with W and Fe/W tips [1]. Fig. 3(b) illustrates a scheme of tunneling from p-GaAs to Fe/W tip.

The averaged  $dI/dV$  curves over the clean part of GaAs(110) with illumination of right and left circular-polarized light obtained with a nonmagnetic W tip and a magnetic Fe-coated W tip are shown in Fig. 4(a) and (b), respectively. It is clear that there exists a dependence of the differential conductivities of GaAs with an Fe-coated W tip on the helicity of illuminating laser light at negative sample bias where partially spin-polarized electrons in the conduction band of p-GaAs tunnel into spin-dependent Fe empty states, while no difference exists in those with a nonmagnetic W tip. This shows that a new channel of tunneling of optically pumped electrons in the conduction band, in

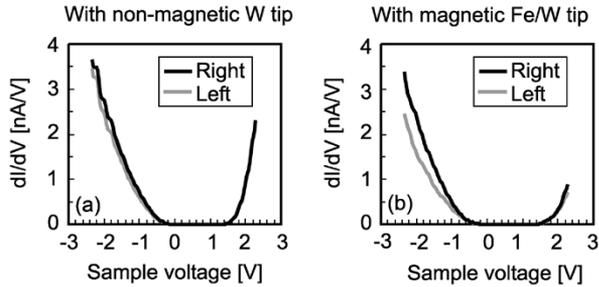


Fig. 4. (a) and (b) show  $dI/dV$  curves obtained with a nonmagnetic W tip and an Fe-coated W tip, respectively, on GaAs surface with illumination of right (black) and left (gray) circular-polarized light at the same set point of  $V_s = +2$  V,  $I = 0.15$  nA.

addition to the tunneling from valence band, is opened in this STS. Since the conduction band is about 1.5 V higher than the valence band, a transition probability from the former is about one order of magnitude larger than that from the latter.

The asymmetry is defined as  $A_{(dI/dV)/T} = [((dI/dV)/T)_{\text{right}} - ((dI/dV)/T)_{\text{left}}] / [((dI/dV)/T)_{\text{right}} + ((dI/dV)/T)_{\text{left}}]$ , where  $((dI/dV)/T)_{\text{right}}$  ( $((dI/dV)/T)_{\text{left}}$ ) denotes the normalized differential conductivity by the transition probability,  $T$ , with illumination of right (left) circular polarized light [2]. This is considered to be equal to  $P_s P_t \cos \theta$ , where  $P_s$  and  $P_t$  are the spin polarizations of tunneling electrons of sample and tip, respectively, and  $\theta$  is the angle between the polarization axes of the apex of the tip and the sample [2], [3]. Circular-polarized laser light incident with an angle of  $75^\circ$  to the surface normal is refracted to  $15^\circ$  to the surface normal inside the GaAs crystal ( $n = 3.7$ ) [6]. Thus, the unit vector in the direction of polarization axes of conduction electrons in p-GaAs(110) can be written as  $\hat{s} = \sin 15^\circ \hat{x} + \cos 15^\circ \hat{z}$ , where  $\hat{z}$  denotes a unit vector of the surface normal, and  $\hat{x}$  and  $\hat{y}$  denote unit vectors perpendicular to the surface normal. The unit vector in the direction of spin at the apex of the tip can be written as  $\hat{t} = \cos \phi \hat{x} + \sin \phi \hat{y}$ . Thus, we get  $\cos \theta = \sin 15^\circ \cos \phi$ , and  $P_s = 0.3$  from the ellipticity of pumping light in GaAs [6].

Due to the uncertainty of angle  $\phi$  of spin direction of a tip apex, asymmetries  $A_{dI/dV}$  obtained with different Fe-coated W tips differ each other. If we assume a maximum observed value of  $A_{dI/dV}$  corresponds to a case for  $\phi = 0$ , we know  $\theta$ . The tunneling transition probability must be independent of the helicity of pumping laser light, but, so far, we have experimental difficulty adjusting the distance between a tip and a sample, which depends on light intensity to get equal transition probability for both helicities.

It should be noted also that in usual SP-STs studies for magnetic samples with Fe-coated W tips, electrons tunnel from the Fermi level of the tip to empty states of a sample in a positive sample bias. In this case, information about spin-polarization of

a ferromagnetic tip at the Fermi level is necessary. It is desirable that the polarization of a spin polarized tip be calibrated in each SP-STs experiment with this method.

## V. CONCLUSION

Holographic TEM indicates the in-plane magnetization at the apex of successful spin-polarized Fe/W tips. The differential conductivities obtained with Fe-coated W tips on a GaAs surface depend on the helicity of the illuminating laser light at a negative sample bias where partially spin-polarized electrons tunnel into spin-dependent Fe empty states, while there is no dependence of those with nonmagnetic W tips.

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