Nb-PtNi-Nb Josephson Junctions Made by 3D FIB Nano-Sculpturing

T. Golod\textsuperscript{a}, H. Frederiksen\textsuperscript{b} and V. M. Krasnov\textsuperscript{a}

\textsuperscript{a} Department of Physics, Stockholm University, Albanova University Center, SE-10691 Stockholm, Sweden
\textsuperscript{b} MC2, Chalmers University of Technology, SE-41296 Göteborg, Sweden
E-mail: taras.golod@physto.se

Abstract.

We use Focused Ion Beam (FIB) for fabrication of nano-scale Superconductor-Ferromagnet-Superconductor (SFS) Josephson junctions, aiming to achieve a uniform, mono-domain state in the F-layer within the junction. We employ a \(\text{Pt}_{1-x}\text{Ni}_x\) alloy, characterized by the perfect solubility of the two components, for obtaining a homogeneous diluted ferromagnet. We perform a systematic analysis of both chemical composition, and ferromagnetic properties of \(\text{Pt}_{1-x}\text{Ni}_x\) thin films for different \(\text{Ni}\)-concentrations. The nano-scale homogeneity of the \(\text{Pt}_{1-x}\text{Ni}_x\) films is confirmed by energy dispersive X-ray spectroscopy. The Curie temperature of \(\text{Pt}_{1-x}\text{Ni}_x\) films decreases in a non-linear manner with \(\text{Ni}\) concentration. We observe that the critical current density of \(\text{Nb-Pt}_{1-x}\text{Ni}_x\text{-Nb}\) junctions decreases non-monotonously with increasing \(\text{Ni}\)-concentration: at \(x \approx 30\%\) it exhibits a minimum, which we attribute to switching into the \(\pi\) state as a function of \(\text{Ni}\)-concentration.

1. Introduction

Hybrid SFS devices put strong constrains on the F-layer: technologically the F-layer should be thick enough, \(\sim 20\text{nm}\), to form a uniform Josephson barrier without pin-holes. This in turn requires that the F-layer is made of a weak, diluted F-alloy, to allow a significant supercurrent [1]. Even more requirements are imposed on spin-valve devices, which require monodomain F-components with uniform spin polarization. This can only be achieved by decreasing the size of the F-layers and by using the shape anisotropy. However, this puts strong demands on the nano-scale spatial homogeneity of the F-alloys. Another reason for decreasing the total area of SFS junctions is a very small resistance per unit area, which require SQUID measurements [1].

The \(\text{PtNi}\) alloy is one of the best candidates for the F-material in nano-scale S/F devices because \(\text{Pt}\) and \(\text{Ni}\) form a solid solution in any proportion [2], unlike \(\text{CuNi}\) and many other nickel alloys, which are prone to phase segregation [3]. The onset of ferromagnetism in bulk \(\text{Pt}_{1-x}\text{Ni}_x\) at room \(T\) occurs at \(x \approx 42\%\) [2].

In this work we fabricate nano-scale SFS \(\text{Nb-Pt}_{1-x}\text{Ni}_x\text{-Nb}\) junctions by 3D FIB nano-sculpturing, which allows fabrication of junctions with area down to \(\sim 50\times 50\text{nm}^2\). As F-material we use the \(\text{Pt}_{1-x}\text{Ni}_x\) alloys with \(\text{Ni}\)-concentration ranging from 0 to 60 at.\%. The nano-size of the junctions both facilitates the mono-domain state in the F-barrier and allows measurements with conventional technique due to sufficiently large junction resistance. To characterize the \(\text{Pt}_{1-x}\text{Ni}_x\) thin films, we study the anomalous Hall effect and perform a nano-scale analysis.
of structural and chemical composition of $Pt_{1-x}Ni_x$ thin films by means of energy dispersive X-ray spectroscopy (EDS). We observe that the Curie temperature of $Pt_{1-x}Ni_x$ thin films decreases in a non-linear manner with $Ni$-concentration and that the critical current density of $Nb-Pt_{1-x}Ni_x-Nb$ junctions decreases non-monotonously with increasing $Ni$ concentration, which we attribute to switching into the $\pi$ state [1].

2. Sample fabrication

$Nb/Pt_{1-x}Ni_x/Nb/Pt_{1-x}Ni_x$ multilayers were deposited on oxidized $Si$ wafer in a single vacuum cycle by DC magnetron sputtering. The $Nb$ film thicknesses were 225 and 350 nm and the critical temperatures 8.6K and 8.8K, respectively. The $Pt_{1-x}Ni_x$ layers were deposited at a certain off-axis displacement between the target and the wafer providing the thickness gradient from 20 to 30 nm on the wafer. Therefore, SFS junctions with different $F$-layer thickness, but the same $Ni$-content were cut of from different parts of the same wafer. To vary the composition of the $Pt_{1-x}Ni_x$ alloys, the corresponding number of small rectangles of $Ni$ were_symmetrically attached on top of the $Pt$ sputtering target. Each rectangle covered about 5% of total target area. The intermediate layer of $Pt_{1-x}Ni_x$ was used as the junction barrier, while the upper layer was employed for improving adhesion with bonding contacts.

The deposited multilayer was patterned with standard photolithography, $Ar^+$ milling ($PtNi$) and CF$_4$ RIE etching ($Nb$). Six electrodes with the width $\sim$ 6$\mu$m were made on each chip. The rest of processing was performed in situ in a dual beam SEM-FIB (FEI Nova 200).

![Figure 1. FIB image of the junction. (a) top view and (b) side view. The insets show schematic of the sample holder (right of 1(a)), and top (a) and side (b) views of the structure.](image)

For 3D FIB nano-sculpturing [4] we used a custom-built 45$^\circ$ wedge holder, which allowed positioning of the sample both perpendicular and parallel to the FIB, see the right inset in Fig. 1a). First, the electrodes were narrowed to $\sim$ 0.5$\mu$m with a ion beam current of 100 pA, and then to the final size (the junction width) ranging from $\sim$ 80 nm to 300 nm with the ion beam current of 10 pA, see Fig. 1a). The side cuts were made with the ion beam parallel to the sample surface with the ion beam current of 10 pA, see Fig. 1 b). The distance between two side cuts, which determine the junction length, varied from $\sim$ 70 nm to 1500 nm.
3. Characterization of \(PtNi\) thin films and \(Nb - PtNi - Nb\) junctions

Magnetic properties of thin films can be quite different from that for the bulk material. Therefore, to characterize the F-barrier, we studied thin (50-100 nm) \(PtNi\) films. Fig. 2 a) shows \(T\) dependence of the Hall resistance \(R_H^*\), normalized by the applied perpendicular magnetic field, for films with different \(Ni\) concentration. The \(R_H^*\) was determined as \(R_H^* = \frac{[R_H(H^+) - R_H(H^-)]}{2}\), where \(R_H(H^\pm)\) are Hall resistances for positive and negative field directions. The Curie temperature \(T_{Curie}\) corresponds to the onset of the anomalous Hall effect. Fig. 2 c) shows the Hall resistance as a function of magnetic field at \(T = 2K\). It is seen that for the film with 30\% \(Ni\)–target area, the Hall effect is anomalous with saturation at \([|H| > 0.4T]\). On the other hand, the Hall effect remains normal (i.e., linear in field) for the film with 10\% \(Ni\)–target area, implying that this film remain paramagnetic even at 2\(K\).

To characterize the strength of our diluted F-thin films, in Fig. 2 b) we show the normalized Hall resistance, \(R_H^*/H\), at 4.2\(K\) (squares, left axis) and \(T_{Curie}\), obtained from the onset of \(R_H^*/H\) vs. \((T)\) in Fig. 2 a), (open circles, right axis), as a function of the relative \(Ni\)–target area. As expected, both increase with \(Ni\)–concentration. The \(T_{Curie}\) increases in a non-linear manner with increasing \(Ni\) concentration.

![Figure 2](image)

**Figure 2.** (Color online) (a) normalized Hall resistance vs. \(T\) for different \(Ni\) concentrations. (b) normalized Hall resistance (■) at 4.2\(K\) and \(T_{Curie}\) (○) as a function of \(Ni\) concentration. (c) Hall resistance vs. field.

![Figure 3](image)

**Figure 3.** The EDS measured \(Ni\) concentration of \(PtNi\) films as a function of the relative \(Ni\) target area. The dashed line represents linear fit to measured datas. The inset shows EDS \(Ni\) mapping of \(P_{60}Ni_{50}\) film.

To determine the actual concentration and the nano-scale spatial distribution of \(Ni\) in \(PtNi\) thin films, we use SEM-based energy-dispersive X-ray spectroscopy [5]. It should be noted that the EDS signal for such thin films can be distorted by the signal from the substrate. To exclude this parasitic signal we have studied EDS spectra at different electron energies, which determine the EDS interaction volume and the resolution. It was observed, that at energies above \(~ 10keV\) the result became independent of the energy, as shown in Fig. 3, presumably due to smaller influence of back-scattered electrons from the substrate. Fig. 3 shows the \(Ni\) concentration, measured by EDS, as a function of the relative \(Ni\)–target area. It is seen that the actual \(Ni\) concentration is 1.2 times larger than the relative \(Ni\)–target area, reflecting the difference in effective deposition rates between \(Ni\) and \(Pt\).

SEM-EDS allows also nano-scale mapping of the alloy homogeneity. The inset in Fig. 3 shows such an EDS \(Ni\)–mapping in a window \(60 \times 80 \mu m^2\) (\(Ni\)–signal in white). No visible inhomogeneity could be detected within the given spatial resolution \(~ 50nm\).
Typical $I−V$ characteristics and Fraunhofer $I_c(H)$ patterns of our junctions can be found in Ref.[6]. In Fig. 4 we plot the critical current density $J_c$ versus $T$ (in the semi-logarithmic scale) for junctions with different $Ni$—concentrations. The $J_c(T)$ becomes exponential for high $Ni$—concentration. Such the $T$—dependence is typical for proximity coupled junctions, in which case it is determined by the $T$—dependence of the coherent length $\xi(T)$ in the barrier. If so, $\xi_F$ for our junctions with strong F-barrier should have the $1/T$ dependence, typical for clean normal metals. This is somewhat surprising since $\xi_F$ is expected to have a negligible $T$—dependence in strong ferromagnets, at least in the dirty case [7].

![Figure 4.](image1.png)  
**Figure 4.** (Color online) ln$J_c$ vs. temperature for Ni concentrations corresponding to 0, 15, 40 and 45 % of Ni target area.

![Figure 5.](image2.png)  
**Figure 5.** $J_c$ as a function of Ni concentration. The inset shows $J_c$ vs. barrier thickness for different Ni concentrations.

Fig. 5 shows $J_c$ at the $T = 3.1 − 3.2K$ as a function of Ni—concentration. It decreases from 0 to 30 % of $Ni$—target area. For larger Ni—content (40 and 45 %) the maximum $J_c$ becomes larger, before it becomes unmeasurably small in the 50% case. Such a non-monotonous behavior can be due to switching into the $\pi$ state [1] as function of $Ni$—concentration. At the same time, SFS junctions on the same chip with 40 and 45 % Ni area could exhibit a large spread in $J_c$, as marked in Fig. 5, presumably due to considerable stray fields from the F-layers in the electrodes. The inset of Fig. 5 shows dependence of $J_c$ on the barrier thickness for 10, 15 and 20 % Ni—target areas. The $J_c$ decreases with increasing thickness in all cases.

In conclusion, we fabricated nano-scale SFS Nb − PtNi − Nb junctions with sizes down to $\sim 70 \times 80nm^2$. EDS analysis indicated that the employed diluted PtNi ferromagnetic layer is characterized by excellent homogeneity at the nm scale. Therefore, such junctions may be promising for hybrid S/F spin-valve devices, which require small, mono-domain F-barriers.

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