

Research Article

The First Observation of Metallic Behaviour in $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$

Akitoshi Nakata, Syunsuke Yano, Hiroki Yamamoto, Syunsuke Sakura, Yoshihide Kimishima, and Masatomo Uehara

Department of Physics, Yokohama National University, Yokohama, Kanagawa 240-8501, Japan

Correspondence should be addressed to Masatomo Uehara; uehara@ynu.ac.jp

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In $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ which has basically the same crystal structure and the similar electrical configuration ($\text{Ni}^{+3d^9}/\text{Ni}^{2+3d^8}$ mix valence state) with high- T_c cuprate, it has been found that this material shows metallic behaviour down to about 20 K by intercalation and subsequent deintercalation with sulfur. This is the first observation of the metallic state in this system. It is unclear why sulfur-intercalation and deintercalation induce the metallic state. We speculate that sulfur works as an effective getter for removing the interstitial apical oxygen which impedes the metallic conduction. However, the weak localization of carriers in the NiO_2 planes still remains below 20 K and the localization may be one of the obstacles to occurrence of possible superconductivity.

1. Introduction

$(\text{La, Nd, Sm})_{n+1}\text{Ni}_n\text{O}_{2n+2}$ ($n = 2, 3$) contains 2-dimensional NiO_2 planes in the unit cell. Label “ n ” corresponds to the number of NiO_2 planes in the unit cell. The crystal structure is basically the same with high- T_c cuprate with two and three CuO_2 planes in the unit cell such as $\text{YBa}_2\text{Cu}_3\text{O}_y$ and $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$. Moreover, the formula valence of Ni is +1.33 ($n = 3$) and +1.5 ($n = 2$) consisting of Ni^{+3d^9} and Ni^{2+3d^8} states. In spite of these structural and electrical similarities with cuprate, $(\text{La, Nd, Sm})_{n+1}\text{Ni}_n\text{O}_{2n+2}$ shows neither metallic nature nor superconductivity [1–7]. However, the similarities with cuprate make us expect the occurrence of high- T_c superconductivity by tuning the electronic state appropriately such as carrier-doping and applying pressure.

The theoretical studies for $\text{La}_4\text{Ni}_3\text{O}_8$ ($n = 3$) and $\text{La}_3\text{Ni}_2\text{O}_6$ ($n = 2$) have been performed [7–10]. In $\text{La}_4\text{Ni}_3\text{O}_8$ it has been predicted that [7, 8] at $T > 105$ K the material is in a low-spin state and is metallic but, at $T < 105$ K, becomes in high-spin state and is insulating because of quantum confinement within the NiO_2 -trilayers [8]. By applying a pressure of 5 GPa, it has been predicted that the high-spin state disappears and the system takes the low-spin state, resulting in a metal-insulator transition [7]. The insulating behaviour of $\text{La}_3\text{Ni}_2\text{O}_6$ has also been predicted to be due to quantum confinement within the NiO_2 -double

layers [10]. The electrical resistivity (ρ) of $\text{La}_4\text{Ni}_3\text{O}_8$ under pressure indicates a suppression of the high-spin state and reduction of ρ [11]. However, neither the metallic state nor superconductivity has been observed [11]. Experimentally, this material is an insulator even at $T > 105$ K. Regarding this discrepancy between experiment and theory, Pardo and Pickett interpret that part of the Ni is not in a low-spin state even at high temperature [7]. If all the Ni atoms take low-spin state, an insulator-metal transition will occur and the emergence of superconductivity can be expected.

In our previous chemical pressure experiments for $(\text{La, Nd, Sm})_4\text{Ni}_3\text{O}_8$, the clear correlation between the a -axis length and ρ was observed [12]. $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ has the shortest a -axis length and the smallest ρ at 300 K in $(\text{La, Nd, Sm})_4\text{Ni}_3\text{O}_8$ solid solution. The electrical resistivity measurements under pressure of 0–3 GPa for $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ suggest a suppression of the high-spin state and reduction of ρ down to 10^{-1} (Ohm-cm) at room temperature [13]. However, neither the metallic state nor superconductivity was obtained being the same result as for $\text{La}_4\text{Ni}_3\text{O}_8$. In order to obtain metallic state and superconductivity, the other strategy in addition to the application of pressure is necessary.

In this paper we report that $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ shows metallic behaviour down to about 20 K by intercalation and subsequent deintercalation with sulfur. This is the first observation of the metallic state in this system.

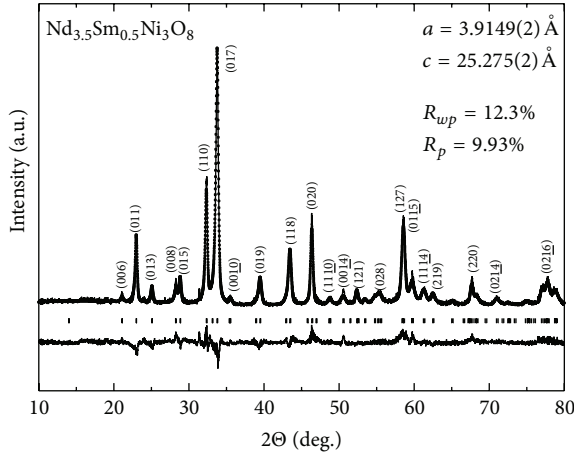


FIGURE 1: XRD data for $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$. Tick marks and bottom solid line are Bragg positions and difference between calculated and observed data, respectively.

2. Experimental

At first $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_{10}$ was synthesized by solid state reaction and the detail was reported in our previous paper [12]. Next, resultant $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_{10}$ was annealed for 5 h at 400°C under flowing hydrogen to reduce $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_{10}$ to $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$.

Sulfur-intercalation was performed in evacuated Pyrex® glass tube together with a 10 weight % sulfur pellet at 400°C for 5 h. Sulfur-deintercalation was performed in tube furnace at 350°C for 3 h under flowing hydrogen gas.

The samples were checked by X-ray diffraction (XRD) using $\text{Cu K}\alpha$ radiation. Rietveld analysis was carried out by using RIETAN-2000 program [14]. The electrical resistivity measurements were performed by conventional four-probe method. Magnetic data were collected by Quantum Design MPMS magnetometer.

3. Results and Discussion

Figure 1 shows the XRD pattern for $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ before sulfur-intercalation. XRD pattern indicates that the sample is almost single-phase. Rietveld analysis was performed by using the crystal model reported previously [3]. Bottom solid line shows the difference between observed and calculated data. From this analysis, the lattice parameters $a = 3.9149(2) \text{ \AA}$ and $c = 25.275(2) \text{ \AA}$ were obtained.

As previously reported, ρ for $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ without sulfur-intercalation shows insulating behaviour, which remains insulating under pressure up to 3 GPa [13]. In order to obtain metallic state, sulfur-intercalation was tried. Assuming that formal valence of sulfur is -2 , the intercalation corresponds to hole-doping. Figure 3 shows the XRD pattern for $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ after sulfur-intercalation. S/N ratio of XRD pattern is not so good because of a small amount of the sample. XRD pattern can be indexed by tetragonal symmetry with $a = 3.8548(8) \text{ \AA}$ and $c = 26.883(9) \text{ \AA}$. Comparing to $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ before sulfur-intercalation, a -axis shrinks

at 1.5% and c -axis expands at 6.4%. There exists $\text{La}_4\text{Ni}_3\text{O}_9$ and this material can be regarded as the compound that an oxygen atom is intercalated in-between sites of NiO_2 layer of $\text{La}_4\text{Ni}_3\text{O}_8$ having the identical structure to $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ [15, 16]. By this oxygen-intercalation for $\text{La}_4\text{Ni}_3\text{O}_8$, a -axis is shrunk at 2% and c -axis is expanded at 5% [15, 16]. The variation of lattice constants of oxygen-intercalation has the very similar tendency with the case of sulfur-intercalation for $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$. From this fact, it is natural that sulfur ions are expected to be intercalated in-between sites of NiO_2 layers. Even though S/N ratio of XRD data is not good due to small amount of sample volume, tentative Rietveld analysis was applied to this sample based on the same crystal model ($I4/mmm$) with $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ plus sulfur ions sitting in-between sites of NiO_2 layers ($4e$ site $(0, 0, 0.06)$). Variable parameters were set to be lattice constants and sulfur occupancy of $4e$ site. From this analysis, it has been found that XRD pattern can reasonably be explained by this model and sulfur content was determined to be $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8\text{S}_{0.7\pm 0.2}$.

By sulfur-intercalation, ρ was decreased from 10^0 ($\text{Ohm}\cdot\text{cm}$) down to 10^{-1} ($\text{Ohm}\cdot\text{cm}$) as shown in Figure 4. However, temperature dependence of ρ remains semiconductive. Although the amount of intercalated sulfur is relatively large (0.7 ± 0.2 at a molar ratio), the reduction of ρ is at most of one order. The superconductivity of this system is thought to appear on the basis of the similarity with the 2-dimensional high- T_c cuprate. Sulfur-intercalation spoils the structural similarity with high- T_c cuprate, even though the system approaches to metallic phase. Therefore, the strategy of sulfur-intercalation by obtaining the metallicity does not seem to be promising.

Next, we performed the sulfur-deintercalation with the condition mentioned in experimental section. Figure 2 shows XRD pattern for $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8\text{S}_{0.7}$ after sulfur-deintercalation. Bad S/N ratio of XRD pattern is attributed to a small amount of the sample. XRD profile seems to return to that of as-synthesized $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$. Therefore, sulfur ions are thought to be successfully deintercalated. Figure 5 shows the temperature dependence of ρ for sulfur-deintercalated $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$. Contrary to expectation, ρ shows metallic behaviour down to 20 K. It is natural to expect that ρ exhibits semiconducting behaviour because the original crystal structure possessing semiconducting nature is recovered. Below 20 K, the electrical conduction is localized and the superconductivity was not observed down to 2 K. The weak semiconducting behaviour below 20 K obeys the 2-dimensional variable range hopping (VRH) model ($\rho \propto \exp(C \cdot T^{-1/3})$, $C = \text{constant}$), reflecting the 2-dimensional intralayer hopping of carriers in the NiO_2 planes. The temperature dependence of $\log \rho$ for sulfur-deintercalated $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ as a function of $T^{-1/3}$ appears to be almost linear below about 20 K as seen in Figure 6. It should be noted that the fitting result was not so bad in 3-dimensional case too (not shown). This might come from a small variation of $\rho(T)$ in the narrow temperature range used by fitting procedure. Otherwise, it might be inherently difficult to strictly appreciate the dimensionality by using VRH model, because in this material three NiO_2 planes are stacked perpendicular to 2-dimensional NiO_2 plane and hence interlayer hopping is also possible.

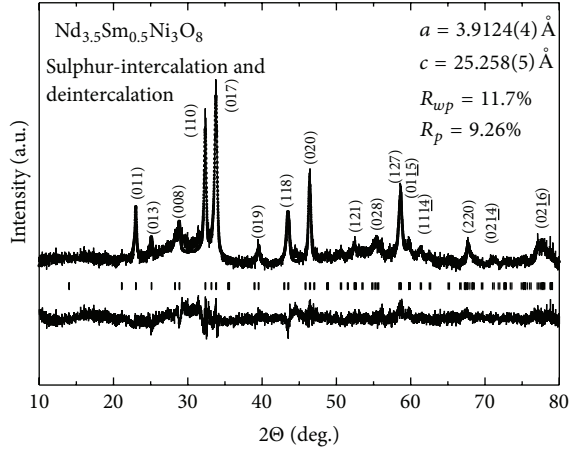


FIGURE 2: XRD data for sulfur-deintercalated $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$. Tick marks and bottom solid line are Bragg positions and difference between calculated and observed data, respectively.

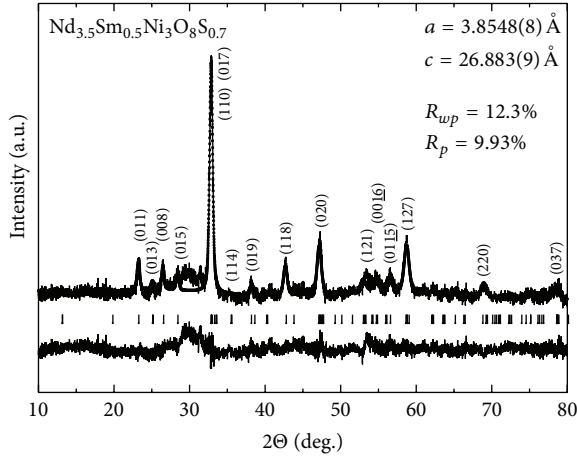


FIGURE 3: XRD data for $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8\text{S}_{0.68}$. Tick marks and bottom solid line are Bragg positions and difference between calculated and observed data, respectively.

The possibility that the metallic behaviour comes from the existence of metallic impurity such as elemental Ni should be considered. Actually very small amount of elemental Ni is detectable in XRD data of Figure 2. In order to know how much elemental Ni exists in the sample, the magnetization measurement as a function of external field was performed at 5 K (see Figure 7). The extrapolated value to 0 Oe of the linear curve fitted to the magnetization data of 15–35 kOe is 1.3 (emu/g). The result means that the sample contains only about 2.2 weight % ferromagnetic Ni. This corresponds to be far below the percolation threshold. Therefore, the metallic nature is thought to be intrinsic.

In order to know what happens by sulfur-deintercalation, Rietveld analysis was tentatively performed, though the quality of XRD data is not so good due to small amount of sample. The best fitting result was obtained with the crystal model in which 10% sulfur ions replace the oxygen site (0, 1/2, 1/4) in block layer and oxygen ions of NiO_2 plane are not replaced

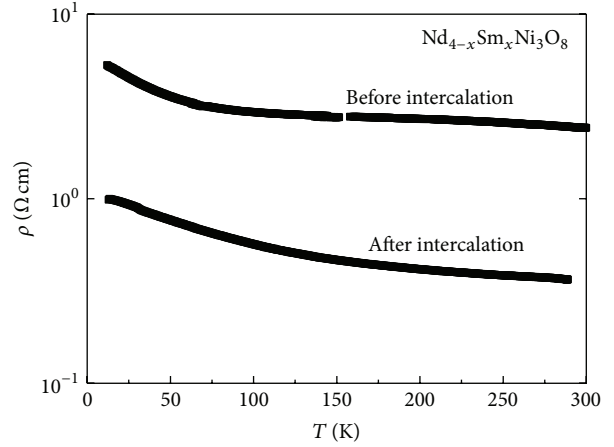


FIGURE 4: Temperature dependence of $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ and sulfur intercalated $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8\text{S}_{0.68}$.

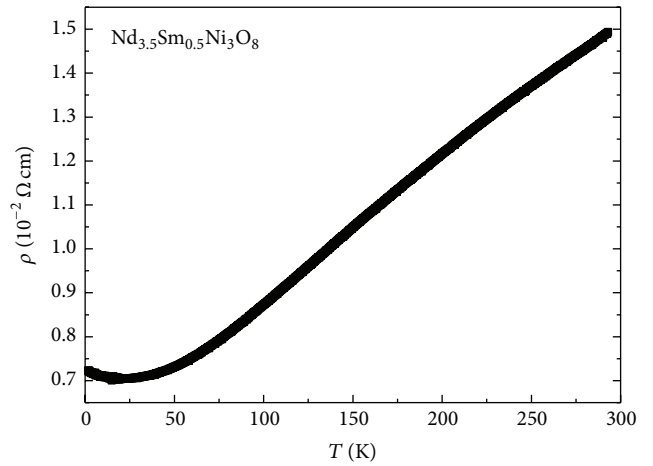


FIGURE 5: Temperature dependence of ρ for sulfur-deintercalated $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$.

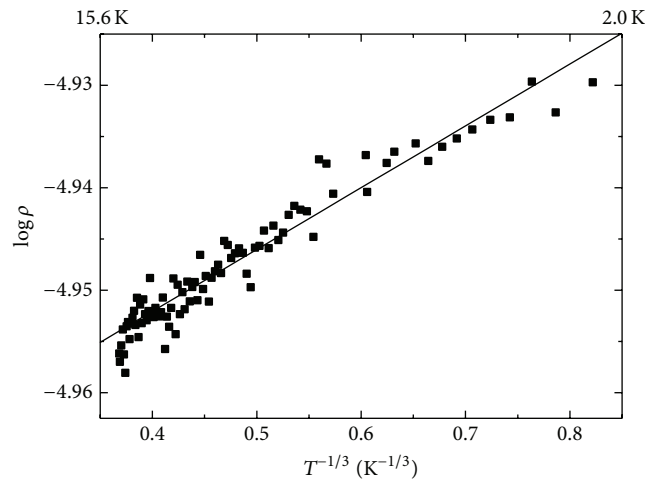


FIGURE 6: $T^{-1/3}$ dependence of $\log \rho$ for sulfur-deintercalated $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$.

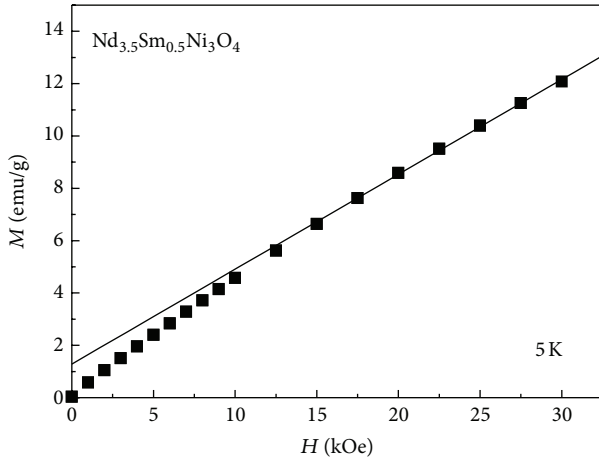


FIGURE 7: Field dependence of magnetization for sulfur-deintercalated $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$.

by sulfur. If sulfur doping to oxygen site really takes place, ionic state of sulfur is thought to be -2 , considering the ionic size matching between sulfur and oxygen. In this case, band filling of the material does not change. However, the replacement of as large amount as 10% sulfur to oxygen site seems to be unreasonable because the change of lattice parameters as mentioned below is very small considering the size difference between sulfur and oxygen. Even if the replacement with sulfur takes place, the amount may be much smaller than 10%. The Rietveld fitting result is shown in Figure 2. The lattice parameters a (c -axis) are shrunk from $3.9149(2) \text{ \AA}$ ($25.275(2) \text{ \AA}$) to $3.9124(4) \text{ \AA}$ ($25.258(5) \text{ \AA}$), comparing the sample without sulfur-intercalation and deintercalation. However, the variations are as small as about 0.1%.

The block layer separating NiO_2 planes is of fluorite type, similar to T' -type high- T_c cuprate. It has been known that T' -type structure tends to incorporate the additional oxygen into the apical oxygen site and this oxygen is a strong obstacle against the superconductivity [17]. We speculate that in $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ the additional apical oxygen exists and the sulfur ions work as an effective oxygen getter for the additional oxygen. Because hydrogen gas is used during sulfur-deintercalation, the hypothesis might be possible as follows: hydrogen chemically reacts with sulfur in the crystal, producing H_2S , and H_2S is going outside the crystal. At the same time, reducing agent H_2S effectively removes the additional apical oxygen with the chemical reaction such as $\text{H}_2\text{S} + \text{O}(\text{apical}) \rightarrow \text{H}_2\text{O} + \text{S}$.

In structurally related compounds T' -type high- T_c cuprate and LaNiO_2 , removal of the additional apical oxygen causes the shrinkage of c -axis length [18–22]. In $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ the shrinkage of c -axis has been observed (from $25.275(2)$ to $25.258(5) \text{ \AA}$) by sulfur-deintercalation. However, the amount of removal of additional oxygen seems to be not enough for inducing the metallicity below 20 K, because the variation of c -axis is smaller (about 0.1%) than that for inducing the metallicity and superconductivity in T' -type high- T_c cuprate. For example of T' -type Sm_2CuO_4 film, the superconductivity can be induced by appropriate reducing annealing,

accompanied by the shrinkage of c -axis with 0.25% [20]. In order to clarify the detailed structural change by sulfur-deintercalation, X-ray diffraction with synchrotron source or neutron diffraction experiments are needed.

4. In Conclusion

In $\text{Nd}_{3.5}\text{Sm}_{0.5}\text{Ni}_3\text{O}_8$ which has basically the same crystal structure and the similar electrical configuration ($\text{Ni}^{1+} 3d^9/\text{Ni}^{2+} 3d^8$ mix valence state) with high- T_c cuprate, it has been found that this material shows metallic behaviour down to about 20 K by intercalation and subsequent deintercalation with sulfur. This is the first observation of the metallic state in this system. It is unclear why sulfur-intercalation and deintercalation induce the metallic state. We speculate that sulfur works as an effective getter for removing the interstitial apical oxygen which impedes the metallic conduction. However, the weak localization of carriers still remains below 20 K in the NiO_2 planes. In order to obtain the metallicity below 20 K and to clarify whether the superconductivity really appears or not, the appropriate annealing condition for removing only interstitial apical oxygen cleanly has to be found out.

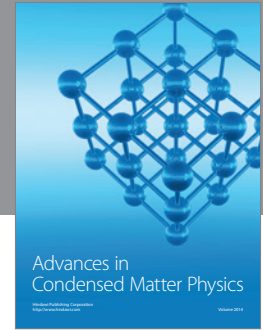
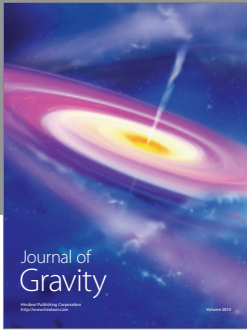
Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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