Selective formation of an ordered variant in Fe$_{45}$Pd$_{55}$ alloy under magnetic field

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Abstract. The effect of magnetic field on microstructure control has been investigated during disorder-order transformation in a bulk single crystal of Fe$_{45}$Pd$_{55}$ alloy, where a two-step ordering heat-treatment has been made to clarify the effect of magnetic field. As a result, single variant of Fe$_{45}$Pd$_{55}$ is obtained when a magnetic field of 10 T is applied only at the early stage of ordering heat-treatment, suggesting that magnetic field is especially effective at the early stage of ordering process.

1. Introduction
Near-equiatomic Fe-Pd alloys undergo a cubic (A1) to tetragonal (L1$_0$) transformation and the ordered tetragonal phase is known to exhibit a large magnetocrystalline anisotropy with an “easy” c-axis [1-3]. In the ordered phase, three crystallographic domains (variants) form equivalently in the absence of external fields. However, under a magnetic field, there arises a difference in magnetic energy among the variants. Therefore, we can expect selective formation of an ordered variant with the lowest magnetic energy under a magnetic field.

In fact, formation of a single variant with its easy axis aligned to the applied magnetic field during disorder-order transformation was firstly reported in Fe-50at%Pd [4-5]. However, how and in which stage magnetic field is effective for the selective formation of specific variant has not been clarified yet. Recently, we have revealed by using a Co-50at%Pt alloy that a single variant of ordered L1$_0$-phase can be obtained by applying magnetic field only in the early stage of ordering heat-treatment [6]. These results suggest that magnetic field is especially effective at the early stage of ordering.

Considering the similarity of crystal structure and magnetic properties between CoPt and FePd, we can expect that the single variant of L1$_0$-type FePd is also obtained by applying magnetic field only in the early stage of ordering heat-treatment.

In the present study, therefore, we have further examined the effect of magnetic field on selective formation of an ordered variant in Fe$_{45}$Pd$_{55}$ alloy by a two-step ordering heat-treatment. The first step of ordering is made under a magnetic field applied in the [001] direction of disordered specimen followed by the second step of ordering at higher temperature under no magnetic field to complete the disorder-order transformation.

2. Experimental procedure
An ingot of Fe-Pd alloy was prepared by arc melting in an argon atmosphere with a nominal concentration of 45at% Fe and 55at% Pd. The ingot was grown into a single crystal by a floating zone...
method. The single crystalline rod was homogenized at 1273 K for 168 h and then quenched in iced-water. Some specimens with all surfaces parallel to \{001\} planes were cut out for ordering heat-treatment and electrical resistivity measurement as shown in figure 1. Three edge directions of cube specimens are defined as X-, Y-, and Z-directions. After cutting the specimens into suitable shapes for each measurement, they were heat-treated at 1173 K for 1 h followed by iced-water quenching to obtain the disordered state.

![Figure 1](image1.png)

**Figure 1.** Schematic illustrations of cube and rectangular parallelepipeds specimens prepared for ordering heat-treatment under magnetic field of $H$ and electrical resistivity measurement.

In order to know the influence of magnetic field on the microstructure formation clearly, we have made a two-step ordering heat-treatment as shown schematically in figure 2. The first step of ordering is made under a magnetic field of 10 T applied in the [001]$_{\alpha 1}$ direction to initiate the ordering under the effect of magnetic field. Then it is followed by the second step of ordering under no magnetic field to complete the ordering process.

In this study, the variant whose easy axis lies in the Z-direction is expressed as Z-variant. The other two variants (X-, Y-variants) are defined in the same way. After the first and second step of ordering, magnetization of the cube specimens was measured at 300 K along the X-, Y-, and Z-directions by a superconducting quantum interference device (SQUID) magnetometer to know the fraction of the variants. To determine the ordering temperature of the two-step ordering heat-treatment, resistivity was measured in heating and cooling processes in disordered and ordered specimens by a direct current four-probe method.

![Figure 2](image2.png)

**Figure 2.** Schematic diagram of the two-step ordering heat-treatment. The bold lines indicate the ordering heat-treatment under a magnetic field and the thin lines indicate the ordering heat-treatment under no magnetic field.

3. Results
Temperature dependence of resistivity during heating and cooling processes of disordered and ordered specimens is shown in figure 3. The dotted curve is resistivity of disordered specimen measured with a heating rate of 100 K/min. The resistivity curve (dotted curve) exhibits a bending at 740 K which
corresponds to the Curie temperature of the disordered phase, \( T_{c}^{(d)} \). The solid curve is resistivity of the ordered specimen measured in the heating process (1 K/min) after a preliminary ordering heat-treatment at 773 K for 24 h. The resistivity heating curve (solid curve) exhibits a bending at 670 K which corresponds to the Curie temperature of the ordered phase, \( T_{c}^{(o)} \). On further heating, the resistivity increases gradually and then decreases sharply due to the transition from the ordered phase to the disordered phase. The order-disorder transformation temperature, \( T_{(o-d)} \) is about 1025 K. The dashed curve is corresponding cooling curve measured from 1200 K to room temperature with a cooling rate of 1 K/min. The disorder-order transformation temperature, \( T_{(d-o)} \) is shown as a sharp increase in resistivity cooling curve (dashed curve) at 955 K. The transformation temperatures are shown with vertical arrows on each curve.

Figure 3. Temperature dependence of electrical resistivity.

Considering the result of resistivity measurement, the conditions of the two-step ordering heat-treatment have been determined: The first step of ordering at 673 K, which is close to the Curie temperature, for 1 h under a magnetic field applied in the [001] direction (Z-direction), followed by the successive second step of ordering at 773 K for 24 h. These temperatures are indicated in figure 2.

Magnetization curves measured at 300 K along the X-, Y-, and Z-directions of as-quenched specimen is shown in figure 4(a). There is no remarkable difference among the curves and they saturate in a low field suggesting that a nearly disordered state is obtained by quenching from 1173 K. Figure 4(b) shows magnetization curves measured after the first step of ordering at 673 K for 1 h under 10 T field. The figure indicates the progress of ordering by this heat-treatment and fraction of the variant whose easy axis lies in the applied field direction (Z-variant) is larger than the fraction of the other two variants (X- and Y-variants) as shown clearly in the inset of figure 4(b). Magnetization curves measured along the X-, Y-, and Z-directions after the second step of ordering at 773 K for 24 h under no magnetic field are shown in figure 4(c). From the figure it is clear that a single variant of Fe_{45}Pd_{55} alloy is obtained.

Similar ordering heat-treatment has been made without applying a magnetic field to assure that selective formation of specific variant is caused by magnetic field. As a result, we have experimentally confirmed that the three variants form nearly equivalently when the first step of ordering is made in the absence of magnetic field.

The result of the two-step ordering heat-treatment described above suggests that selection of variant by magnetic field is essentially determined in an early stage of ordering. That is, during the early stage of ordering in the first step, the fraction of the Z-variant reaches some specific amounts larger than the fraction of the X- and Y-variants due to the application of magnetic field. When this
initial condition is satisfied, magnetic field is not necessary in the following ordering process. For further discussion we need to clarify the microstructure formed at the early stage of ordering under a magnetic field and it is a subject in the future.

**Figure 4.** Magnetization curves measured at 300 K along the X-, Y-, and Z-directions of (a) as-quenched specimen, (b) after the first step of ordering at 673 K for 1 h under magnetic field of 10 T, (c) successive second step of ordering at 773 K for 24 h.

4. Conclusions
We have investigated effect of magnetic field on microstructure formation through disorder (A1)-order (L1₀) transformation by using single crystal of Fe₄₅Pd₅₅ alloy. A single variant is realized when the ordering heat-treatment is made close to the Curie temperature under magnetic field of 10 T applied in the [001] direction of the disordered phase. These results suggest that magnetic field is effective at the early stage of ordering process, as in CoPt previously studied by the authors [6].

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