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Measurement of archeomagnetic intensity of bricks from a Roman ruin in Slovakia

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Abstract: Paleomagnetic intensity of bricks from a Roman ruin in Slovakia dated as 2nd-3rd century A.D. was measured. The Thelliers' method with triple heatings was applied to two bricks named sample A and B. The paleointensity from sample A was measured at the Toyo University and National Institute of Polar Research. The results from the two laboratories agreed roughly with the average of $60.7 \pm 7.6 \mu T$. The paleointensity from sample B was $51.4 \pm 15.0 \mu T$, which was measured at the Toyo University. The magnetic properties suggest that the larger scatter of sample B compared with sample A is possibly caused by heterogeneous oxidization created during the baking process of the sample B brick.

1. Introduction

Constituent furnaces and fireplaces excavated at the ruins can provide information of the geomagnetic field when they were heated. Bricks and pottery baked in the furnaces also can provide geomagnetic information when they were made. Archeomagnetic information reveals secular variation of the geomagnetic pole position and field intensity.

The compilation of archeomagnetic data shows a significant scatter (e.g. Kono *et al*, 1986). The aim of this study is to better understand the cause of the paleointensity data scatter. Prior to this study, paleointensities from Roman bricks were measured at the Geophysics Institute of Slovak Academy of Sciences (SAS) (Gregorova, 1998). In this study, paleointensities of the same samples were measured at the Toyo University (Toyo) and National Institute of Polar Research (NIPR) to compare the results from different measurement systems. At the same time, measurement of the hysteresis loops and thermal magnetic analyses (Js-T curves) were also performed at Toyo to study the magnetic properties of the samples.

2. Samples

Two red bricks were sampled from an ancient ruin called Iza near Komarno (latitude 47° N, longitude 18° E), Slovak Republic. The ruin was built in the 2nd-3rd century A.D. during the Roman Empire along the Danube River. Now it has collapsed completely. Two bricks named sample A and B were collected from the central part of the ruin. The bricks were divided into two pieces and one set was sent to Japan. Cylindrical specimens of one-inch diameter and one inch long, which is the typical size for a paleomagnetic study, were taken from the samples. Samples A and B provided five and three cylindrical specimens, respectively that were measured at Toyo. Another three specimens from sample A were measured at NIPR.

3. Experiment

First, natural remanent magnetization (NRM) was measured for each specimen. For the paleointensity measurement, the successive triple heating Thelliers' method (Thellier and Thellier, 1959) refined by Coe (Coe, 1976a, b) was applied to the specimens. Although double heating is usually performed in the original Thelliers' method, triple heating is used for its higher precision analysis.

The procedure of the successive triple heating method is as follows.

Step 1. Demagnetization of NRM at T_1.
- Heating at T_1 for 20 minutes in the air.
- Cooling to room temperature in a non-magnetic field.

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- Remanent magnetization measurement.
- Magnetic susceptibility measurement.

Step 2. Thermal remanent magnetization (TRM) acquisition to the +X direction at T_1.

- Heating at T_1 for 20 minutes in the air.
- Cooling to room temperature in a field of 50 μT toward the +X direction.
- Remanent magnetization measurement.

Step 3. TRM acquisition to the −X direction at T_1.

- Heating at T_1 for 20 minutes in the air.
- Cooling to room temperature in a field of 50 μT toward the −X direction.
- Remanent magnetization measurement.

Steps 1 to 3 were repeated progressively in steps up to 700 °C. The thermal remanent magnetization (TRM) acquired at steps 2 and 3 should be the same in an ideal case. Thelliers' method relies on the law of additivity of partial TRM (pTRM). This law states that if a sample is heated up to a temperature below the Curie point, only grains with blocking temperatures below that temperature will be affected. A pTRM check was performed on some specimens. In a pTRM check, a pTRM at some lower temperature is reintroduced after the NRM measurement at a higher temperature. The remeasured TRM should give the same value as the first measurement. However, heating often causes the alteration of magnetic minerals and results in erroneous data.

At Toyo, a horizontal type thermal demagnetizer with a three-layered μ-metal shield is used. Both heating and cooling are performed in the μ-metal shield. The residual magnetic field intensity is less than a few nT. The samples are kept at a programmed high temperature for 20 minutes then cooled for 20 minutes with a fan.

A horizontal type thermal demagnetizer at NIPR has a three- and five- layered shield at the furnace and cooling chamber, respectively. The residual magnetic field intensity is less than 20 nT in the cooling chamber. When the samples are cooled, they are moved from the furnace to the cooling chamber. The samples are kept at a programmed high temperature for 20 minutes in the furnace and cooled for 20 minutes in the cooling chamber with a fan.

A vertical type thermal demagnetizer at SAS has three component Helmholtz coils to reduce the geomagnetic field. The residual field intensity may be a few nT at the heating area. The samples are kept at a programmed high temperature for 20 minutes and cooled to room temperature overnight without a fan.

Alternating-field (AF) demagnetization, thermal demagnetization, thermomagnetic analysis (Js-T curve), and hysteresis parameters were obtained from extra specimens to confirm the stability of NRM and determine the magnetic minerals that carry the NRM.

4. Results

The NRM intensities measured at Toyo and NIPR are shown in Table 1. Specimens of sample A have a rather stronger NRM than sample B.

The AF demagnetization curves of NRM on the Zijderveld diagrams (Zijderveld, 1967) are shown in Figure 1. Both samples A and B are stable against AF demagnetization. This means they have or contain little secondary magnetization. The specimen from sample B indicates a higher median destructive field (MDF). It may contain magnetic grains of a higher coercivity than the specimen from sample A.

The Js-T curves were obtained in an external magnetic field at 1.0 T both in a vacuum (10^{-4} Pa) and in the air. Specimens from samples A and B showed similar Js-T curves in both conditions. Curie temperatures of samples A and B are estimated from these curves. They are listed in Table 2.

For example, the Js-T curves of specimens from sample A are shown in Figure 2. The Js-T curve in a vacuum is extremely irreversible with a Curie point at 540°C(Fig. 2(a)). Magnetization appears at about 540°C during cooling. It gradually increases to a value three times as large as the original Js at room temperature. On the other hand, Js-T curves in the air indicate that magnetization during cooling are smaller than those during heating (Fig. 2(b)).

Hysteresis parameters of samples A and B were measured before and after heating for Js-T curves. The experiments were performed as follows. First, hysteresis parameters of one specimen from each sample were measured at room temperature. Next, it

Table 1 NRM intensity. Specimens beginning with A are from sample A and beginning with B are from sample B.

specimen	NRM (Am^2/kg)
A1(Toyo)	1.92E-04
A2(Toyo)	1.92E-04
A3(Toyo)	1.00E-04
A4(Toyo)	1.99E-04
A5(Toyo)	2.14E-04
B1(Toyo)	5.31E-05
B2(Toyo)	6.94E-05
B3(Toyo)	8.47E-05
A2-1(NIPR)	1.62E-04
A2-2(NIPR)	1.42E-04
A3-1(NIPR)	8.41E-05

Table 2 Curie temperature. Tc: Curie temperature, vac.: heating in a vacuum, air: heating in the air.

specimen	Tc vac.(℃)	Tc air(℃)
A	535	537
B	501	548

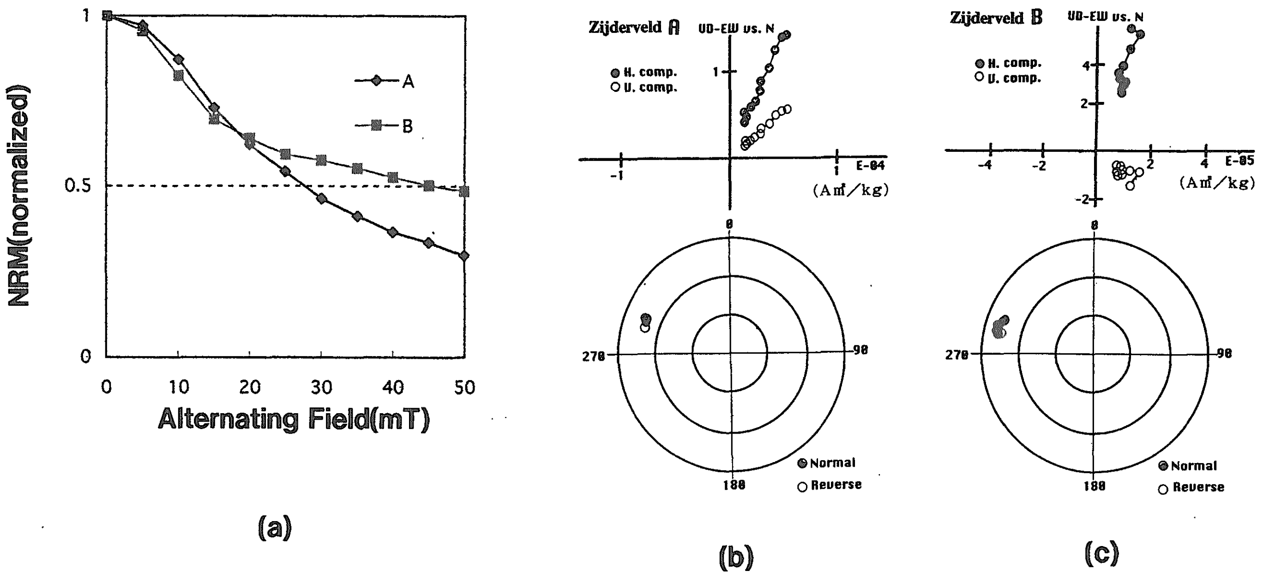
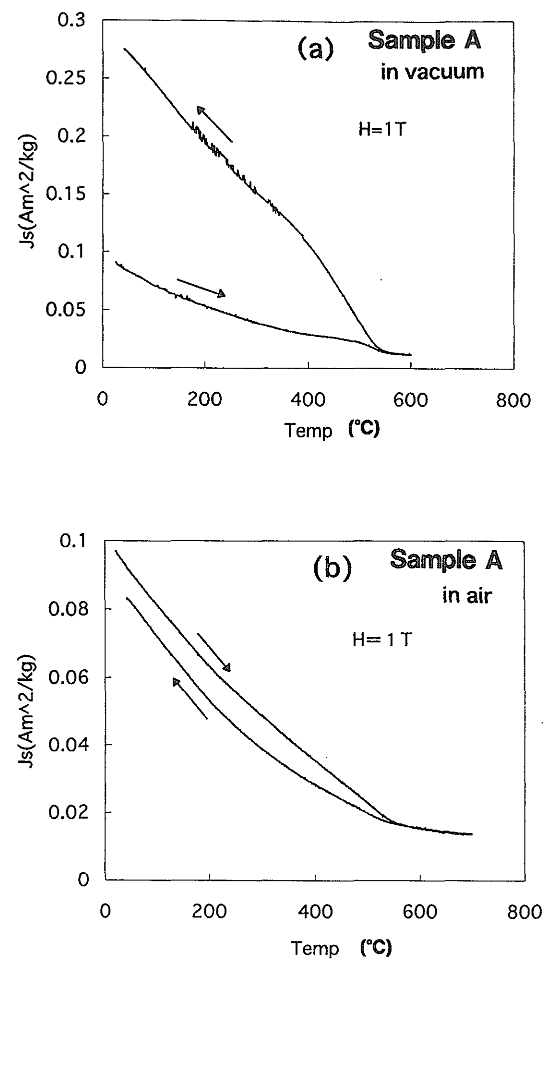


Fig. 1 Alternating field (AF) demagnetization of natural remanent magnetization (NRM).
(a) Stepwise AF demagnetization to 50 mT. NRM is normalized to an initial value. Sample B has a higher coercivity than sample A.
(b) Zijderveld projection and directional change of sample A. Samples are without orientation so directions are relative.
(c) Zijderveld projection and directional change of sample B. Samples are without orientation so directions are relative.



was heated in the air for obtaining the Js-T curve. After, hysteresis parameters of the same specimen were measured. The specimen was named as A-air or B-air. In the same way, a different specimen from the samples was heated in a vacuum for obtaining the Js-T curve after the first measurement of the hysteresis parameters. The hysteresis parameters of the specimen were measured again. The specimen was named as A-vac or B-vac. The hysteresis parameters before heating are listed in Table 3.

The changes of hysteresis loops before and after heating of sample B is shown in Figure 3. The hysteresis loops before and after heating in a vacuum are shown in (a). Similarly, the hysteresis loops before and after heating in the air are shown in (b) with magnified hysteresis loops around the origin. The hysteresis parameters were explained in (c).

The ratios of hysteresis parameters changed from

Fig. 2 (a) Js-T curves in a vacuum (around 10^{-4}Pa). It has a Curie point at around 540°C during heating and cooling. Saturation magnetization became approximately three times as large as that before heating, which implies that hematite was reduced to magnetite whose saturation magnetization is much larger than hematite.
(b) Js-T curve in the aor. Saturation magnetization became approximately 20% smaller than that before heating, which implies that magnetite was partially oxidized to hematite.

before to after heating. The ratios of remanent coercivity (Hrc) to coercivity (Hc) and saturation remanence magnetization (Jr) to saturation magnetization (Js) are plotted in Figure 4. After heating, Hrc/Hc became smaller for every specimen, although the parameters before and after heating are scattered.

The initial magnetic susceptibility was measured at room temperature before the Thelliers' method. Then, susceptibility was measured at room temperature after each stepwise heating in the Thelliers' method (Fig. 5). The specimens of samples A and B show a depression around 350°C–400°C and a small peak around 500°C–600°C. The susceptibility was normalized to the initial value. The decrease of susceptibility began around 350–400°C, which implies that iron sulfides were altered by heating.

In the experiment of the triple heating Theiliers' method, acquired pTRMs of the +X and −X directions were measured. For example, thermal demagnetization of NRM and laboratory acquired pTRM of specimen A1 from sample A are shown in Figure 6. The laboratory acquired pTRMs in the +X and −X directions were almost the same intensity. Other specimens also indicated no significant difference.

Table 3 Hysteresis parameters before heating. Js: Saturation magnetization, Jr: Saturation remanence, Jm: Maximum saturation magnetization, Hc: Coercivity, Hrc: Remanent coercivity. Specimen names with "vac" or "air" mean that the specimens for heating in a vacuum or in the air for future experiments.

specimen	Js E-02 (Am^2/kg)	Jr E-03 (Am^2/kg)	Jm E-02 (Am^2/kg)	Jr/Js	Jm/Js	Hc (mT)	Hrc (mT)	Hrc/Hc
A vac.	4.70	11.40	7.76	0.24	1.65	3.00	60.00	20.00
A air	5.77	6.65	9.64	0.12	1.67	3.10	54.00	17.40
B_1 vac.	8.92	6.92	13.65	0.08	1.53	2.40	120.20	50.10
B_2 vac.	6.27	11.10	12.10	0.13	1.93	2.70	58.00	21.50
B air	8.04	6.13	14.31	0.08	1.78	3.00	125.70	41.90

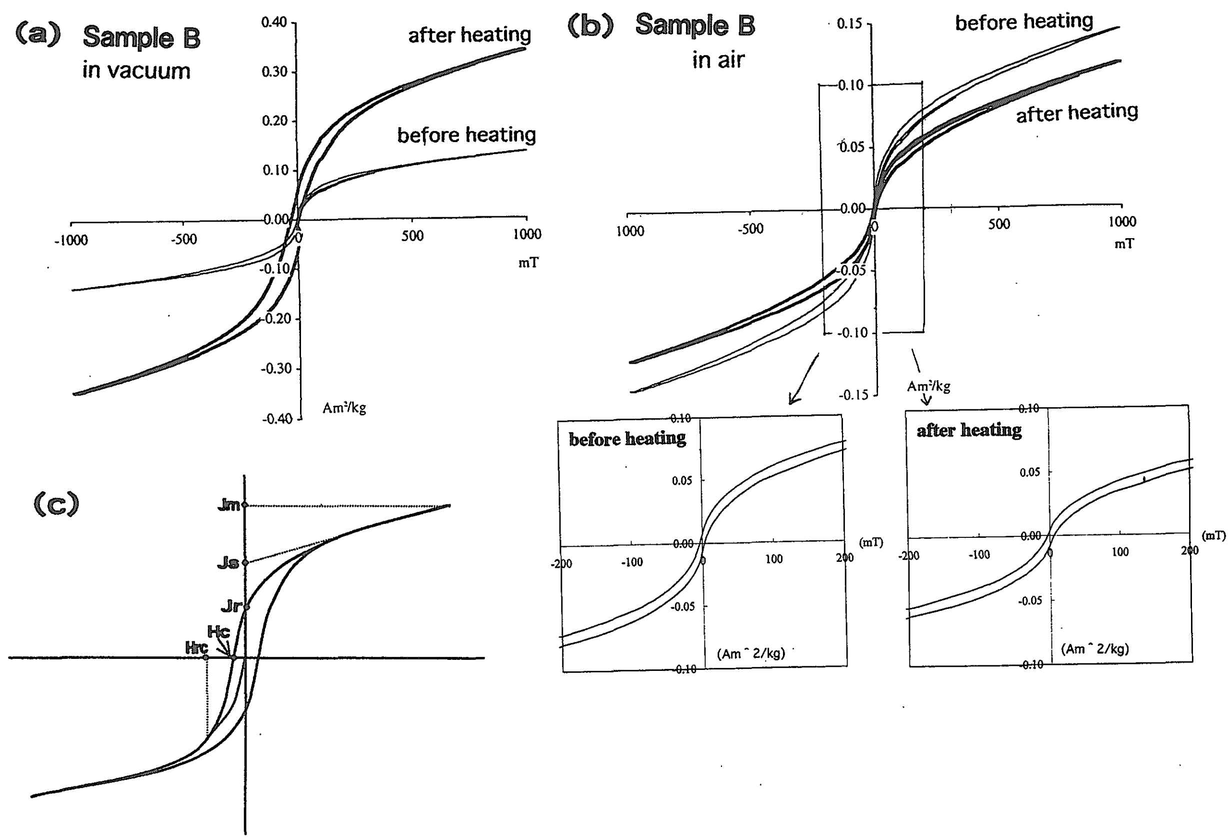


Fig. 3 Change of hysteresis loops of sample B before and after heating. (a) Hysteresis loops in a vacuum. (b) Hysteresis loops in the air with magnified loops around the origin. (c) Explanation of hysteresis parameters.

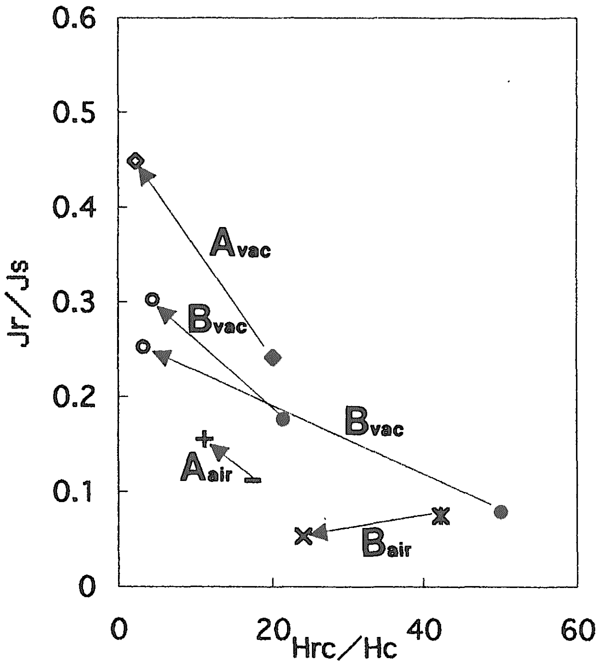


Fig. 4 Change of Jr/Js and Hrc/Hc ratios before and after heating.
"vac" means heating in a vacuum (around 10^{-4}Pa) and "air" means heating in the air.

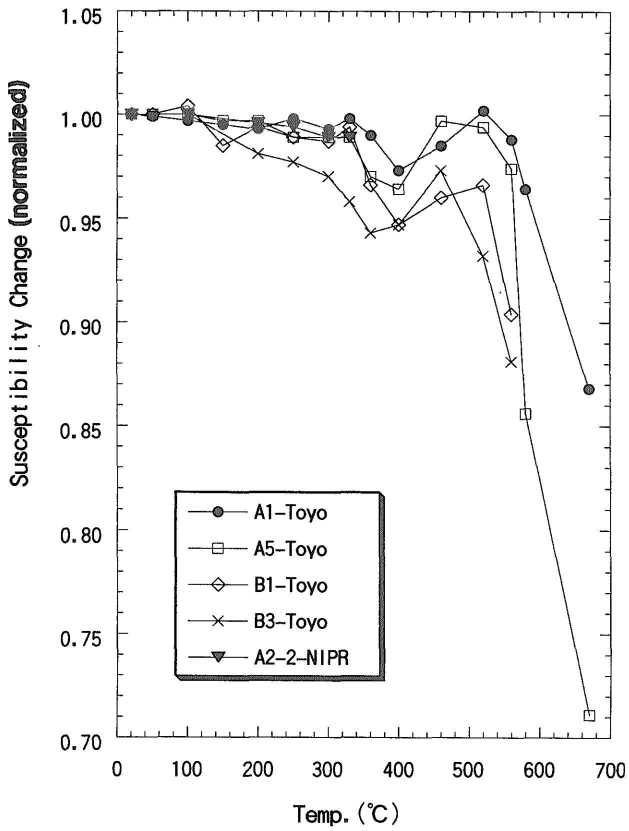


Fig. 5 Susceptibility changes during Thelliers' experiments. ● : Specimen A1 measured at Toyo. □ : Specimen A5 measured at Toyo.◇ : Specimen B1 measured at Toyo.× : Specimen B3 measured at Toyo. ▼ : Specimen A2-2 measured at NIPR.

Three diagrams of each specimen are shown in Figure 7. They are the (a) Arai diagrams (Arai, 1963), (b) thermal demagnetization of NRM and laboratory acquired pTRM and (c) relative magnetic directions on equal area projection changes of NRM and TRM during the triple heating Thelliers' experiment obtained at Toyo and NIPR. The pTRM is the average of the +X and −X directions.

A slight peak of NRM is recognized around 50°C-100°C in six specimens. They are specimen A3 (Fig. 7-3(b)), specimen A4(Fig. 7-4(b)), specimen A5 (Fig. 7-5(b)), specimen A3-1(Fig. 7-7(b)), specimen B2 (Fig. 7-10(b)) and specimen B3 (Fig. 7-11(b)). NRM below 100°C is possibly affected by secondary magnetization.

The NRM-pTRM ratio should remain constant throughout the experiment. The absolute value of the gradient on the Arai diagram indicates the ratio between the paleomagnetic field and laboratory field. To calculate the line gradient from the Arai diagram of each specimen (Fig. 7-1(a) to Fig. 7-11(a)), each line was fitted to the data from 100°C to 350°C-400°C. The data below 100°C are affected by secondary magnetization, and the alteration of magnetic minerals around 350°C-400°C was recognized in the susceptibility changes. The data at 100, 150 and 200 °C of specimens A1, A2 and B1 at Toyo (Fig. 7-1, Fig. 7-2 and Fig. 7-9) were rejected because of the incorrect calibration of the magnetometer. Specimens A2-1 and A3-1 at NIPR (Fig. 7-6 and Fig. 7-7) have no data above 330°C because of mechanical trouble. Specimen A2-2

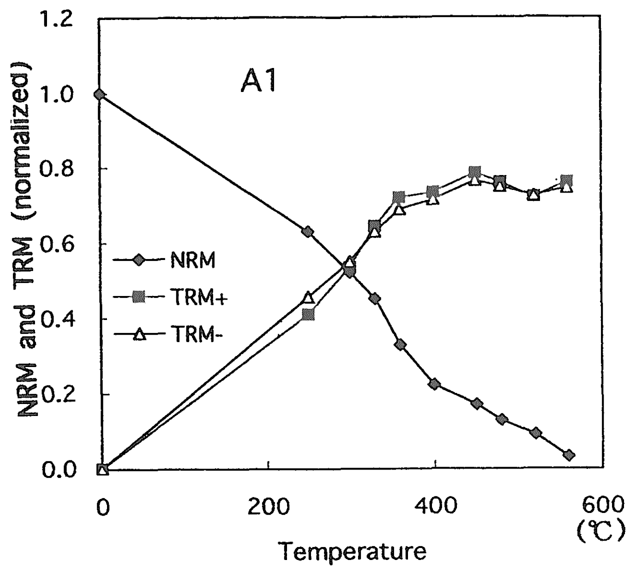


Fig. 6 Thermal demagnetization of NRM and laboratory acquired pTRM of specimen A1 from sample A were measured at Toyo.
◆ : NRM intensity, ■ : acquired pTRM of +X direction, △ : acquired pTRM -X direction. NRM and pTRM are normalized to an initial value of NRM.

at NIPR (Fig. 7-8) was measured after the magnetometer was repaired.

The calculated paleointensity values are shown in Table 4. The paleointensity was calculated by multiplying the absolute value of the gradient by the laboratory field. The laboratory field of Toyo and NIPR was simply considered to be 50μT.

The same samples were measured at SAS. Samples A and B provide four and six specimens, respectively that were measured. The result of sample A was 63.5±10.8μT and sample B was 65.5±10.6 μT (Table 5) (Gregorova, personal communication). The present geomagnetic field of SAS was considered to be 49.7μT.

5. Discussion

The paleointensities of samples A and B were plotted with the change of the geomagnetic field intensity in the period from 4400 B.C. to the present in the Czech and Slovak Republics (Fig. 8). They are consistent with the previous study (Bucha, 1965).

The paleointensities were calculated from the NRM-pTRM relation in the Arai diagram using the data between 100°C and 350°C or 400°C. The depression around 350°C-400°C in the susceptibility curves were apparently caused by the alteration of Fe-Ti oxides. The scatter of the paleointensity values was possibly caused by differences in the magnetic properties of each specimen.

The magnetic carriers of samples A and B are considered to be hematite and magnetite, and the contribution of hematite is more in sample B than sample A. These assumptions are suggested by the following results.

(1) Hysteresis loops indicate high Hrc, suggesting the significance of hematite as a carrier of remanent magnetization. Sample B has an especially higher Hrc than sample A on average.
(2) The Js-T curve shows that magnetic minerals were slightly oxidized from titanomagnetite to hematite during heating in the air. On the other hand, heating in a vacuum resulted in a significant reduction from hematite to magnetite. Hysteresis loops also indicate the same tendency.
(3) The NRM intensity of sample B is smaller than

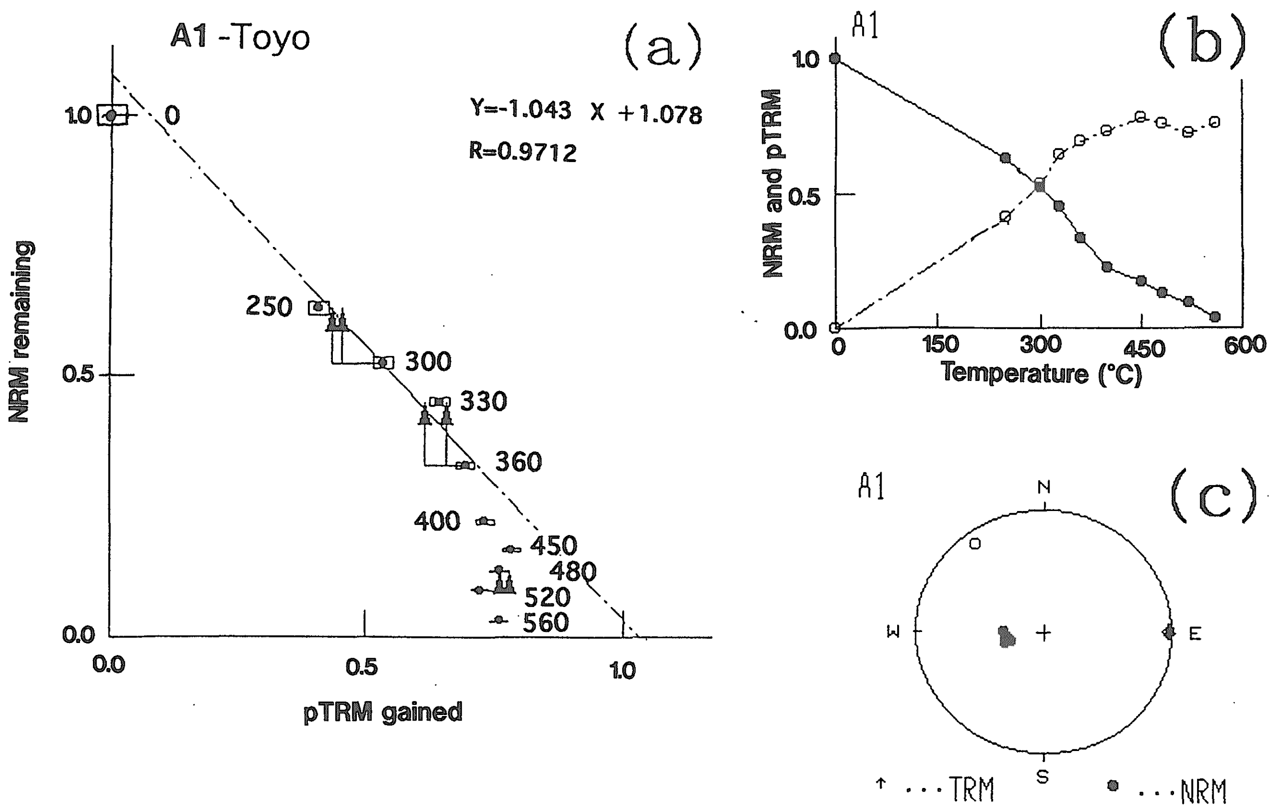


Fig. 7-1 A1 at Toyo.
Fig. 7-1～11 Diagrams of the Thelliers' method for determining absolute paleointensities from each specimen from sample A or B. Figures 7-1 to 7-11 indicate the results of each specimen.
(a) Arai diagram of NRM component remaining versus pTRM acquired for each demagnetization temperature. NRM and pTRM are normalized to an initial value of NRM. Arrows show "pTRM checks".
(b) Thermal demagnetization of NRM and acquisition of pTRM. Solid circles indicate NRM and open circles indicate pTRM.
(c) Change of relative direction of NRM and pTRM with temperature. (Samples are not oriented.)

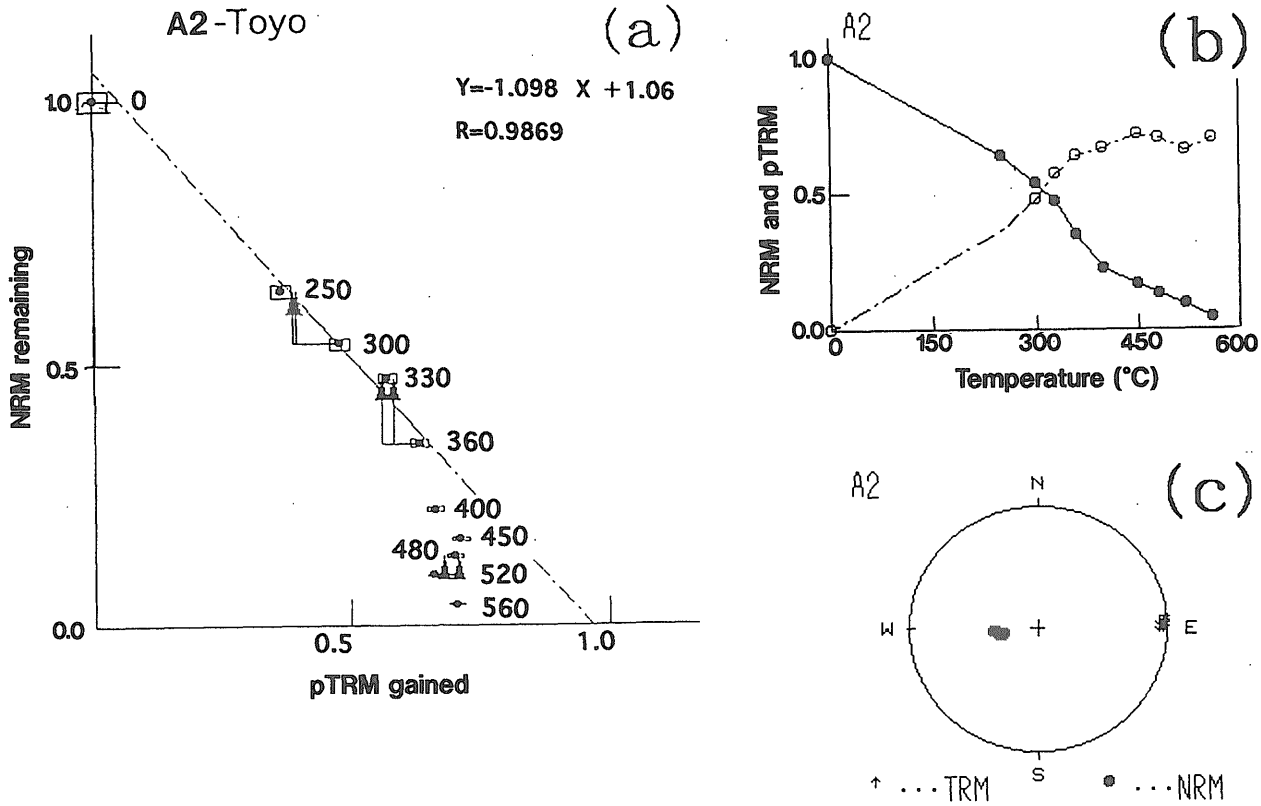


Fig. 7-2 A2 at Toyo.

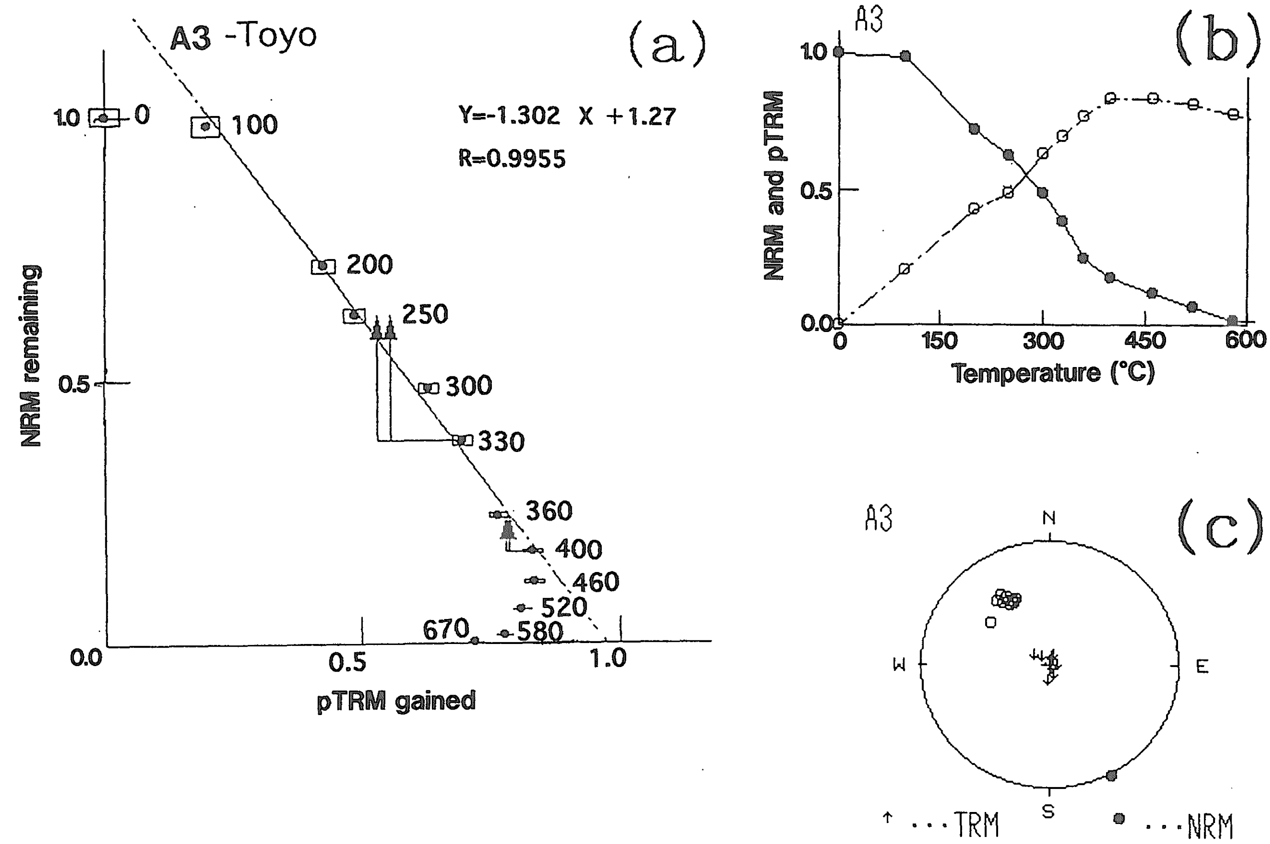


Fig. 7-3 A3 at Toyo.

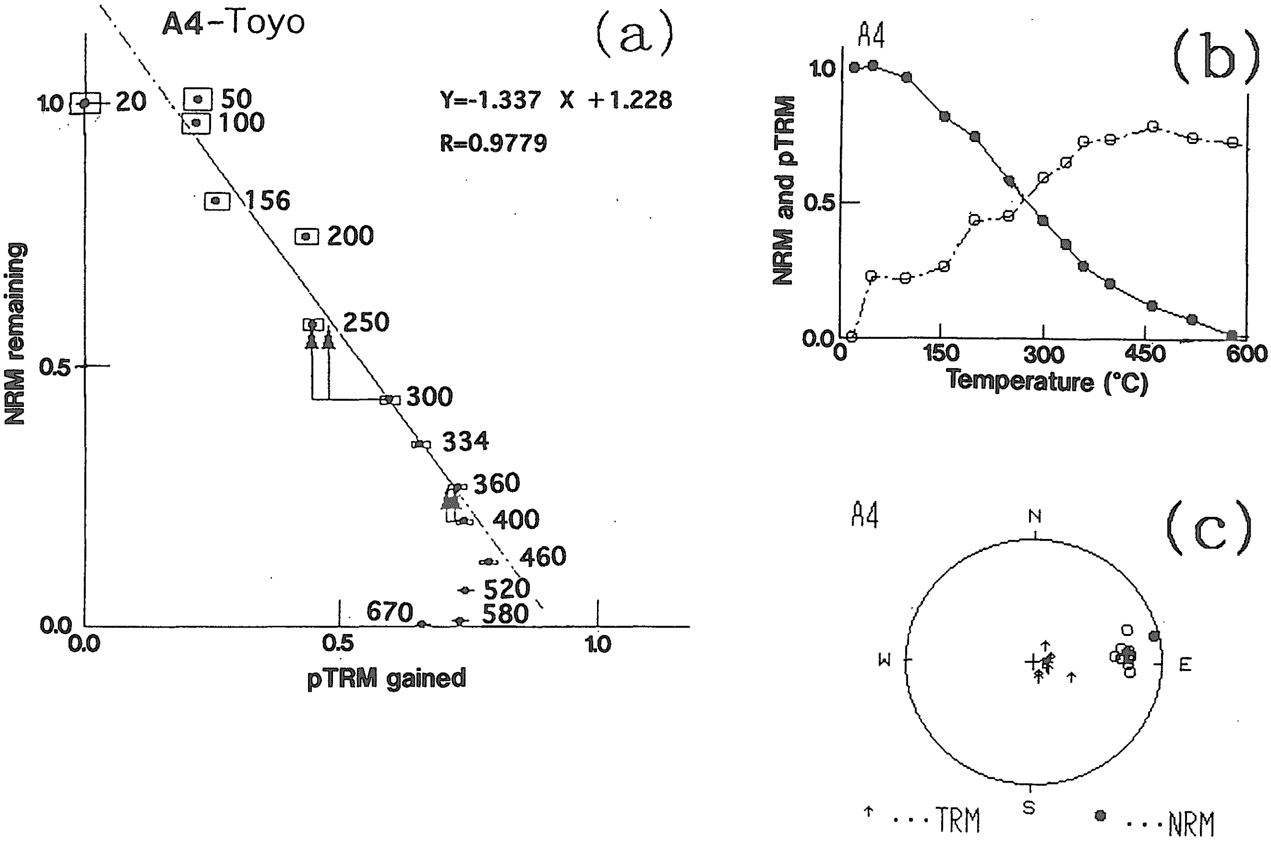


Fig. 7-4 A4 at Toyo.

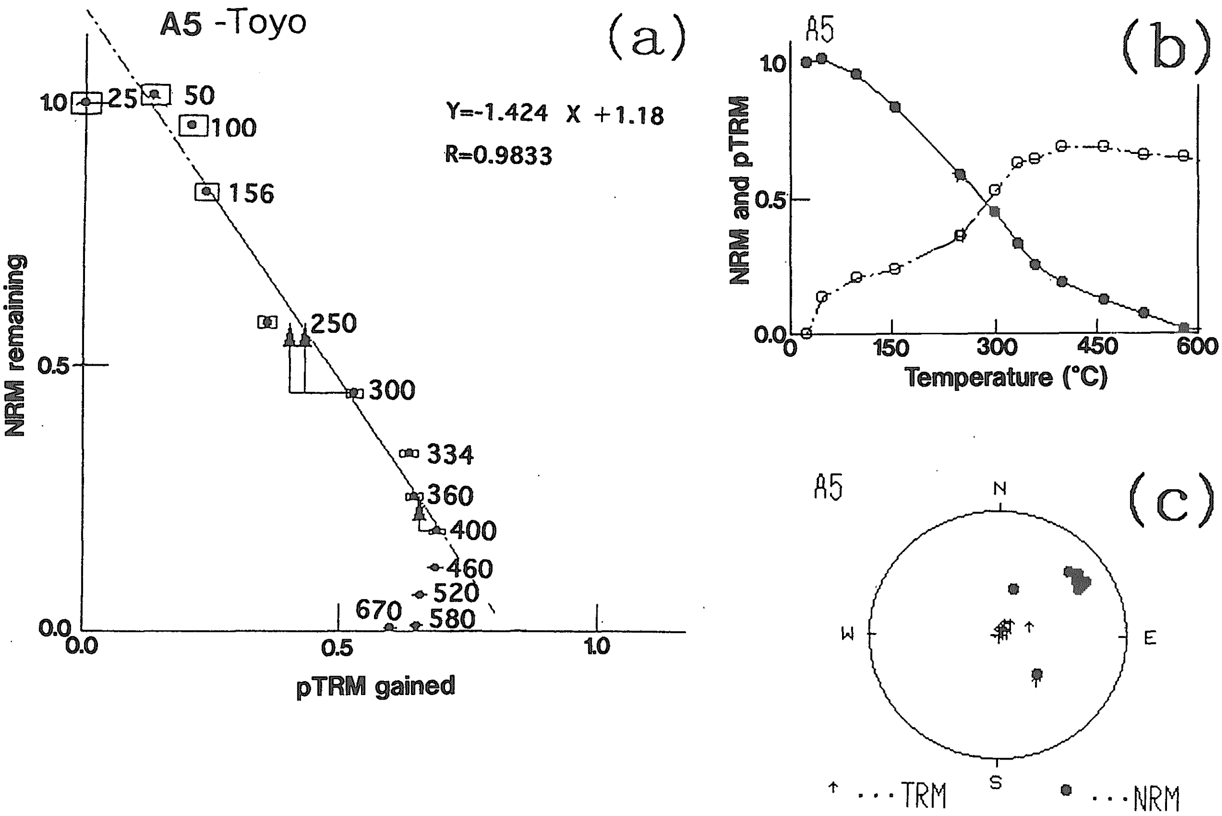


Fig. 7-5 A5 at Toyo.

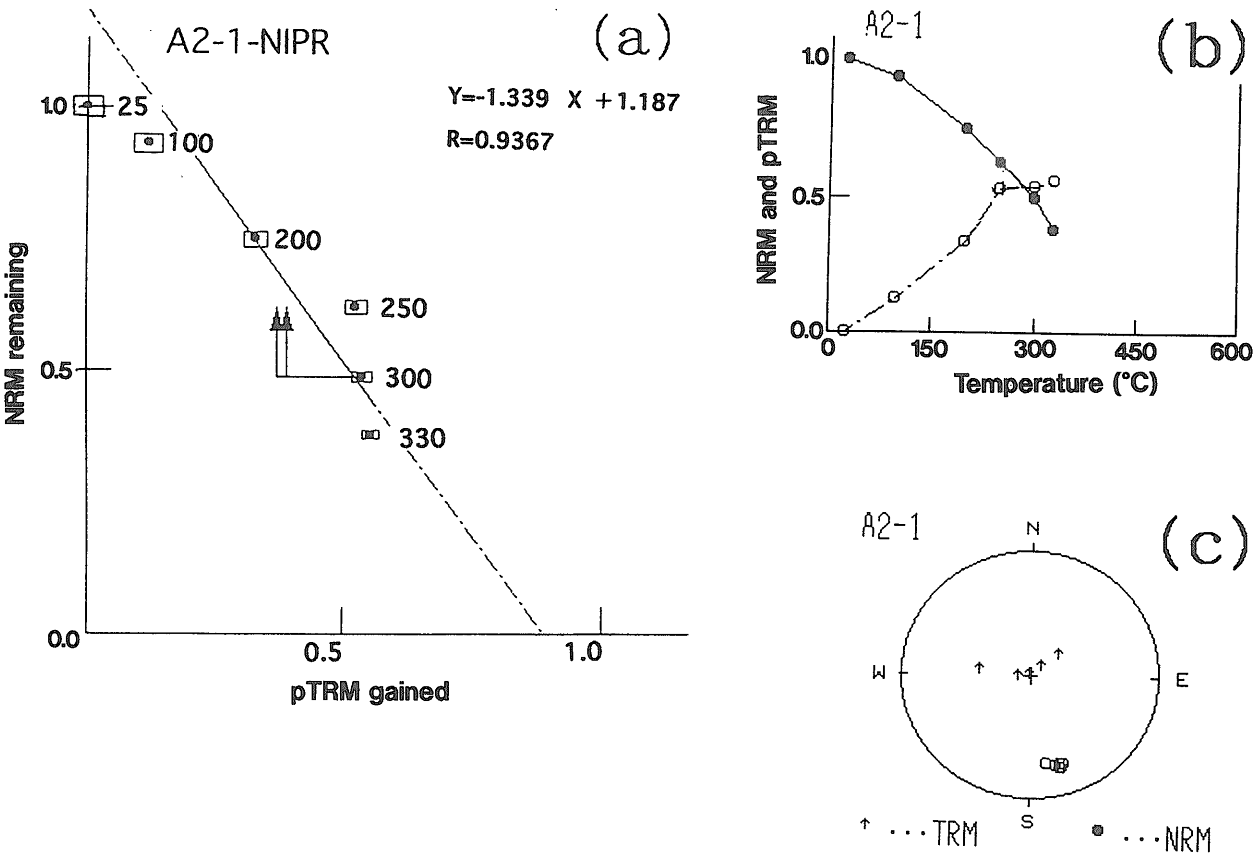


Fig. 7-6 A2-1 at NIPR.

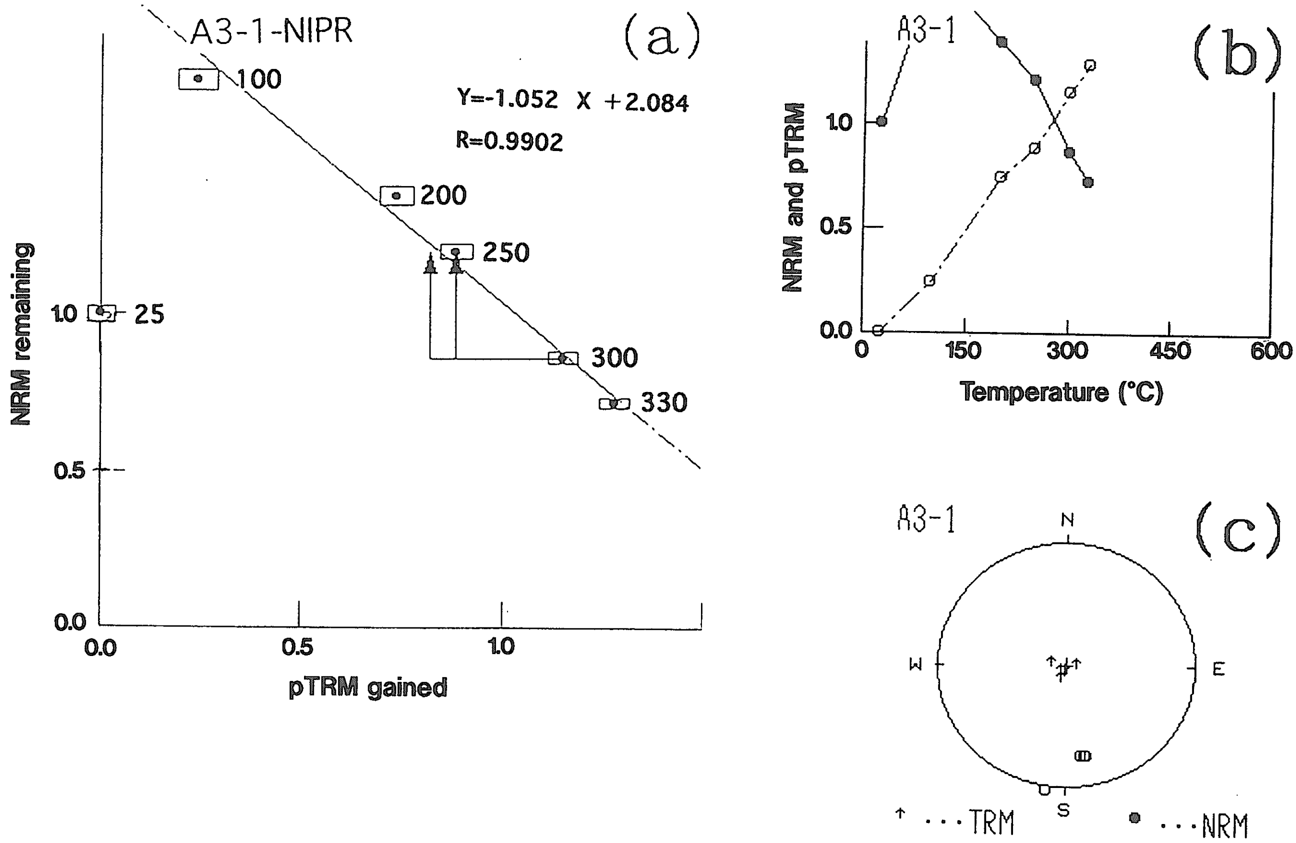


Fig. 7-7 A3-1 at NIPR.

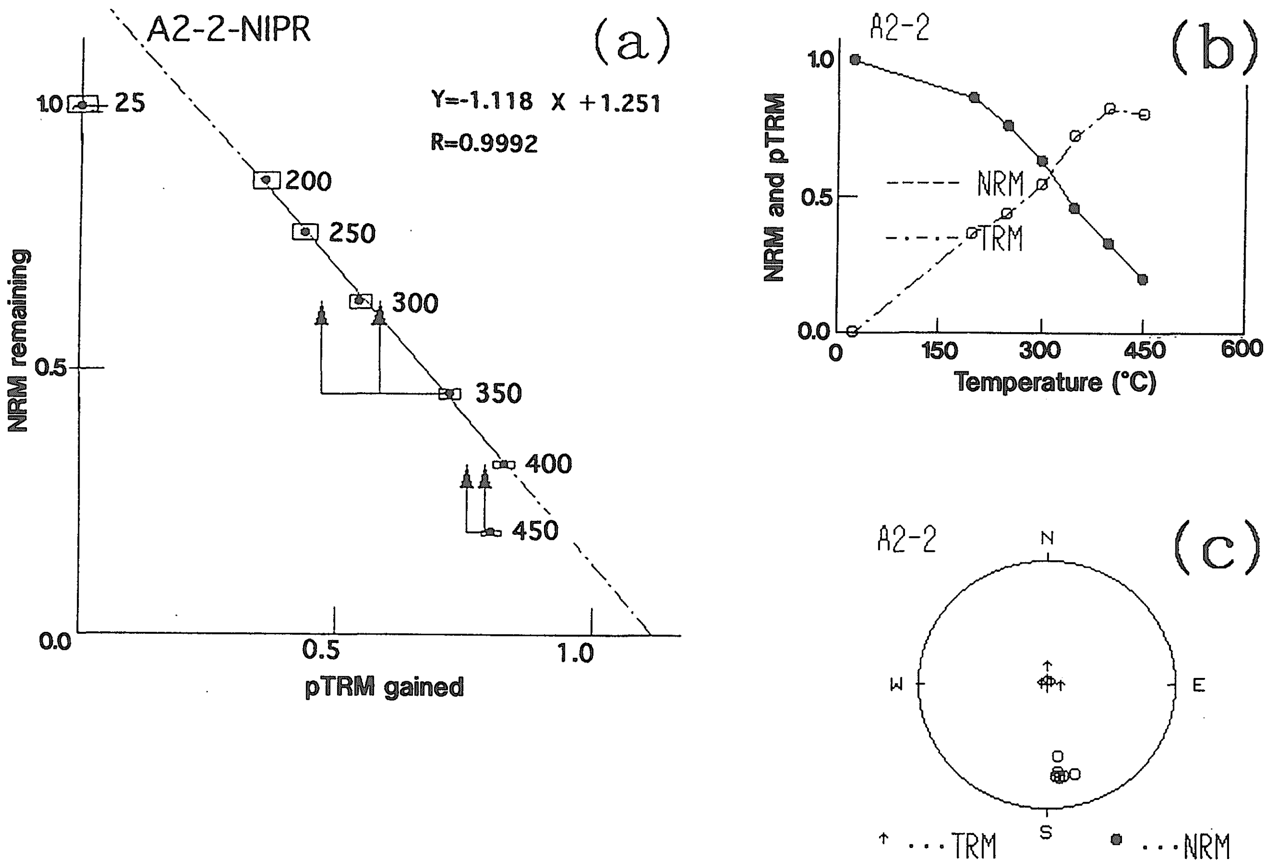


Fig. 7-8 A2-2 at NIPR.

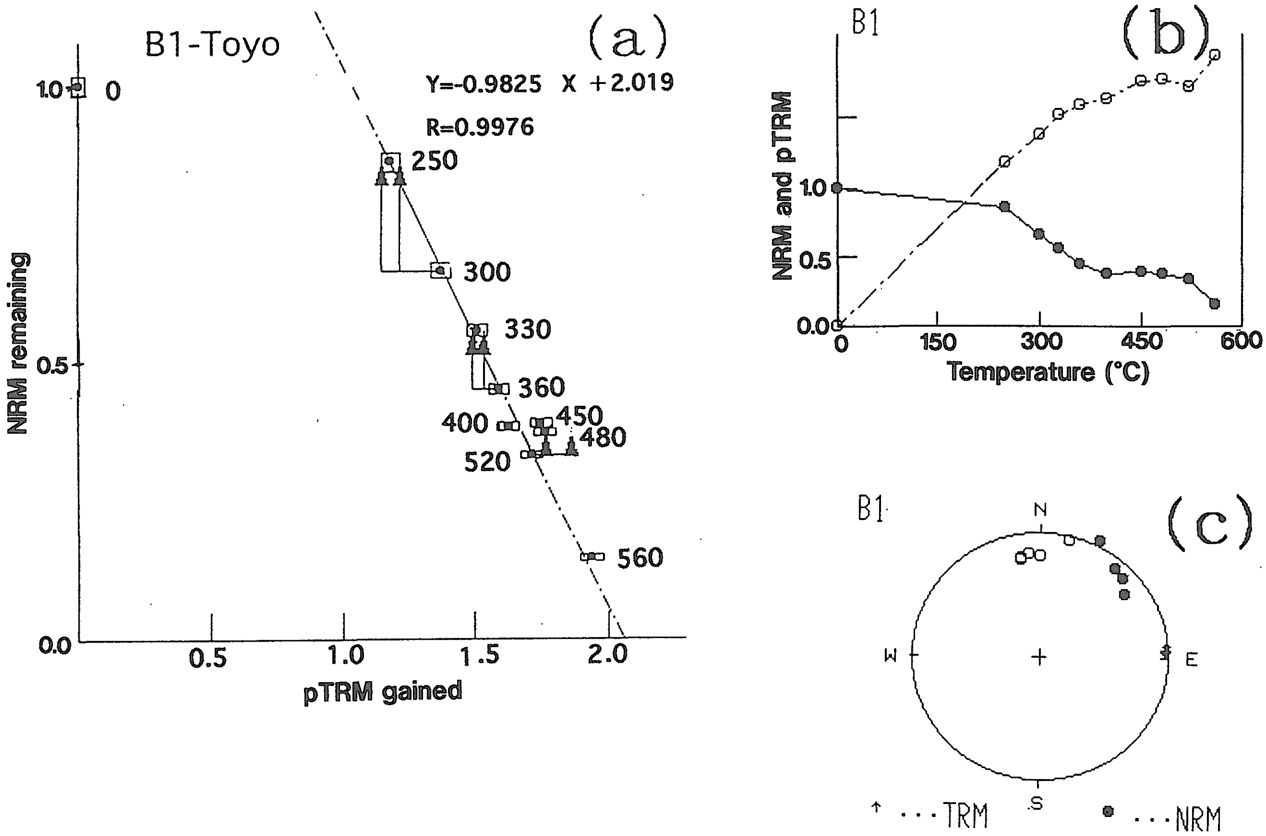


Fig. 7-9 B1 at Toyo.

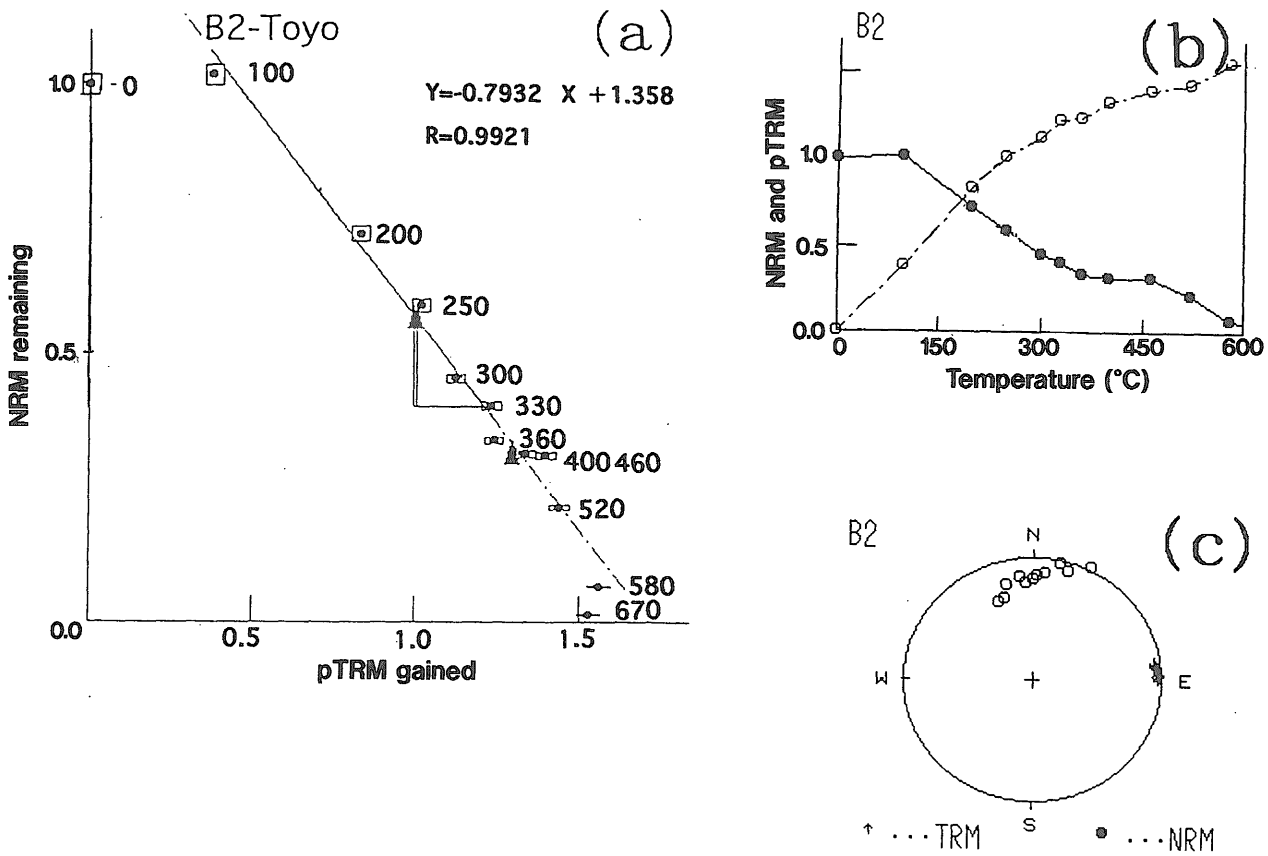


Fig. 7-10 B2 at Toyo.

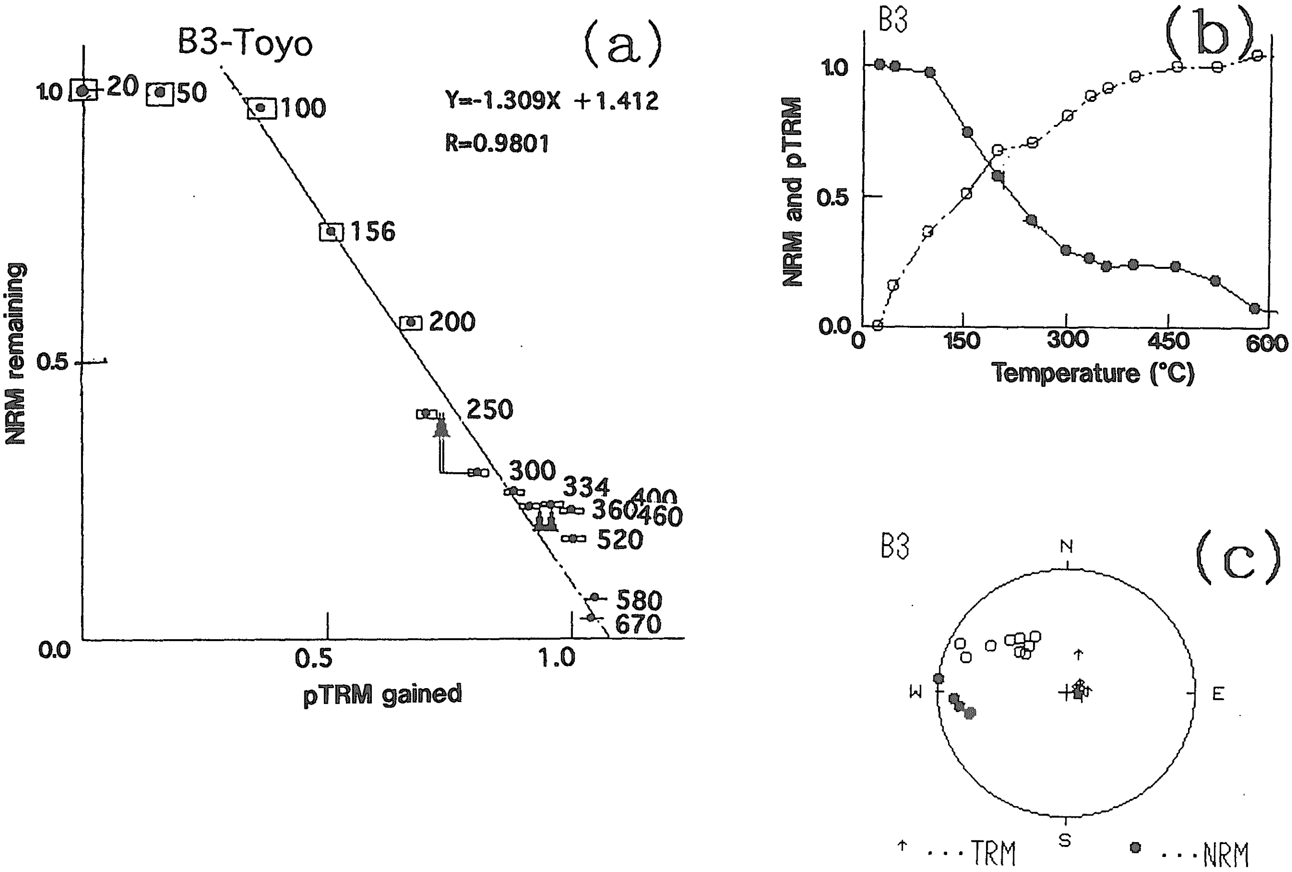


Fig. 7-11 B3 at Toyo.

Table 4 Paleointensity obtained from Arai diagrams is shown in Figure 7.

Paleointensity

specimen	paleointensity (μT)	gradient	lab. field (μT)	calculated data (point)	correlation R^2
A1-Toyo	52.2	1.04	50.0	250-360℃ (4)	0.971
A2-Toyo	54.7	1.09	50.0	250-360℃ (4)	0.977
A3-Toyo	65.1	1.30	50.0	100-360℃ (6)	0.996
A4-Toyo	66.9	1.34	50.0	100-360℃ (7)	0.978
A5-Toyo	71.2	1.42	50.0	156-400℃ (6)	0.983
A2-1-NIPR	67.0	1.34	50.0	100-300℃ (4)	0.983
A3-1-NIPR	52.6	1.05	50.0	100-330℃ (5)	0.986
A2-2-NIPR	55.9	1.12	50.0	200-400℃ (5)	1.000
B1-Toyo	49.1	0.98	50.0	250-360℃ (4)	0.998
B2-Toyo	39.7	0.79	50.0	250-360℃ (6)	0.992
B3-Toyo	65.5	1.31	50.0	100-360℃ (7)	0.980
Aver.-A	60.7±7.6				
Aver.-B	51.4±15.0				

Table 5 Paleointensity obtained from measurements at SAS. (After Gregorova, personal communication)

Paleointensity measured at SAS

specimen	paleointensity (μT)	gradient	lab. field (μT)	calculated data (point)
A1-SAS	69.1	1.39	49.7	100-300℃(4)
A2-SAS	69.1	1.39	49.7	100-300℃(4)
A3-SAS	46.7	0.94	49.7	100-300℃(4)
A4-SAS	65.1	1.31	49.7	100-300℃(4)
B1-SAS	73.6	1.48	49.7	50-400℃(6)
B2-SAS	53.2	1.07	49.7	50-450℃(7)
B3-SAS	69.6	1.40	49.7	50-400℃(6)
B4-SAS	67.6	1.36	49.7	50-450℃(7)
B5-SAS	61.1	1.23	49.7	50-400℃(6)
B6-SAS	68.1	1.37	49.7	50-400℃(6)
Aver.-A-SAS	63.5±10.8			
Aver.-B-SAS	65.5±10.6			

(after Gregorova, personal communication)

that of sample A. It is possibly because of the difference in oxidization.

The NRM intensity and hysteresis parameters show a larger scatter in sample B than sample A.

When the bricks were initially baked sufficiently in a furnace, most part of the titanomagnetite was oxidized into hematite. The red color of the samples indicates the dominance of hematite. It is suggested that sample B was not heated homogeneously when the bricks were initially baked in the furnace. Insufficient heating of the bricks sometimes makes some differences in magnetic properties (Bucha, 1965).

6. Conclusions

Paleointensity of sample A measured at Toyo and NIPR was 60.7±7.6μT while sample B measured at Toyo was 51.4±15.0μT. The results at SAS were 63.5±10.8 μT for sample A and 65.5±10.6 μT for sample B. The results of sample A were in good agreement at the three institutes in spite of different measurement systems, but sample B results were scattered. The deviations and scattered results of B

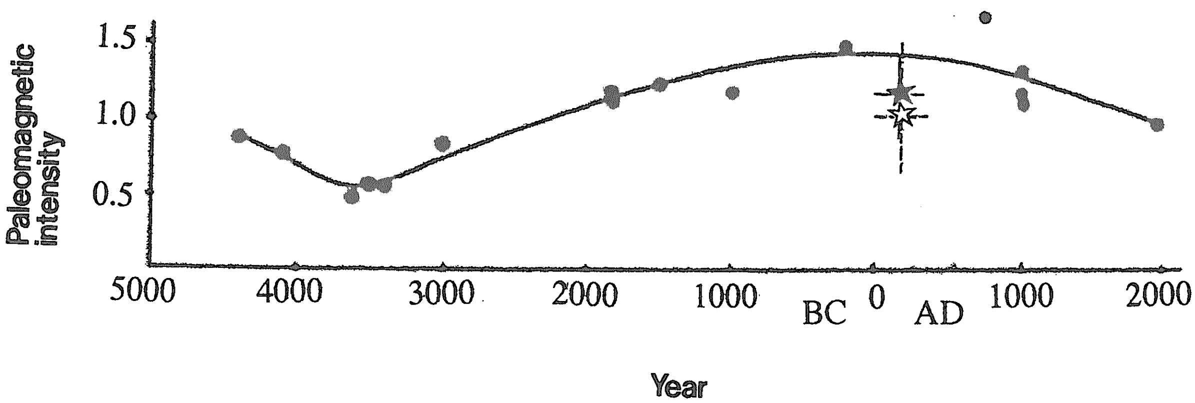


Fig. 8 Changes of the paleomagnetic field intensity in the Czech and Slovak Republics. Paleomagnetic field is normalized to the present field intensity. (after Bucha, 1965 ★: sample A, ☆: sample B)

are possibly caused by heterogeneously baked samples and not by the different measurement systems. Several specimens should be measured in order to enhance the reliability of the paleointensity data.

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スロバキアのローマ遺跡から得られたレンガ片の古地球磁場強度測定

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要　旨

スロバキアのローマ遺跡から得られた AD2～3 世紀と考えられている 2 つのレンガ片，サンプル A および サンプル B を用いて古磁場強度測定を行った．測定には 3 回加熱式テリエ法を用い，サンプル A は東洋大学と国立極地研究所で比較測定を行った．またサンプル B は東洋大学で測定を行った．その結果，サンプル A の示す古地球磁場強度は 2 つの実験室での結果がほぼ一致し，平均 60.7μT，分散 7.6μT となった．サンプル B は平均 51.4μT，分散 15.0μT となった．サンプル B の分散がサンプル A に比べ大きいのはサンプル B がレンガとして焼かれた時，内部の酸化度が非一様であった可能性がある．