Fission Track Age of Miocene Kn-3 Tuff in Central Japan: Towards Better Age-Control on Magneto-Biostratigraphic Time Scale

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A fission track age for a Miocene tuff (Kn-3) in the late Cenozoic sedimentary sequence in the Boso Peninsula, central Japan, was determined in an effort to gain an age control for the biozone boundary. The obtained age of 15.0 ± 0.5 Ma (error: 1σ) represents the N.9/N.10 boundary of the planktonic foraminiferal zones, as the Kn-3 Tuff is located near the first occurrence of Globorotalia peripheroacuta which defines the N.9/N.10 boundary. Our age determination supports most of the recent estimates for the N.9/N.10 biozone boundary which until now has not been dated directly, but inferred based on speculative assumptions.

1. Introduction

The integrated time scale is indispensable in the determination of geologic ages of strata based on the microfossils present. High resolution of the biostratigraphic zonations enables us to understand the geologic history in greater detail, whereas biostratigraphy alone only indicates stratigraphic relations but not a numerical age for a particular unit. Therefore, it is necessary to combine biostratigraphy with magnetostratigraphy when constructing a time scale. Advances on an integrated stratigraphic time scale is dependent on the analysis of deep sea core, while datable volcaniclastic layers are rare within deep sea sediments. Magnetostratigraphy provides worldwide time-horizons, however it only shows the digital signals of normal and reversed polarities. It is thus desirable to determine reliable biostratigraphic, magnetostratigraphic and geochronologic data along the same section in order to establish a high-resolution integrated stratigraphic time scale. In this paper, we discuss the newly obtained fission track age of the Miocene pumice tuff (Kn-3) in the marine sequence of the Boso Peninsula, central Japan, which gives better age-control for the N.9/N.10 boundary of the planktonic foraminiferal zones. Estimated ages for the N.9/N.10 boundary in the proposed time scales are also discussed.

2. Problems of the N.9/N.10 Boundary

Berggren et al. (1995) revised the Neogene time scale from which the numerical age of each biozone can be estimated. The reliability of the geochronometric ages is based on an evaluation of sea-floor magnetic anomalies. The geomagnetic polarity sequence was established primarily from data from the South Atlantic using a combination of finite rotation and averages of anomaly spacings from stacked profiles projected onto a synthetic sea-floor spreading flow line. The geomagnetic time scales were then generated by fitting a spline function to a set of some age calibration points and the zero-age ridge axis to the composite polarity sequence (Cande and Kent, 1992, 1995; Wei, 1995).

Cande and Kent (1992) assigned an age of 14.8 Ma to the younger end of Chron C5Bn (C5Bn(0.0)) based on radioisotopic age constraints correlated to the N.9/N.10 foraminiferal zone boundary (Berggren et al., 1985b). These age constraints were derived from strata in Japan (Tsuchi et al., 1981) and Martinique (Andreieff et al., 1976). In Japan, Tsuchi et al. (1981) estimated the age of the N.9/N.10 boundary as 14.6
Fig. 1. An age estimation of the first appearance datum (FAD) of the genus *Orbulina* in Japan, of which the upper and lower limits were defined in the Kii Peninsula and Hokuriku district, respectively (modified from Tsuchi, 1983). The other important datum levels of planktonic microfossils have been estimated using deep sea core (DSDP Site 298). The age of the *Orbulina* datum (15.5 Ma) was used as a calibration point for estimating the other datum ages. The stratigraphic positions of each level on the core can be regarded to represent each age, assuming a constant sedimentation rate.
Ma, which is defined by the first appearance datum (FAD) of *Globorotalia peripheroacuta* (Blow, 1969). However the premise of this age is tenuous.

The age of the FAD of *Grt. peripheroacuta* was estimated based on its stratigraphic position in a deep-sea core (DSDP Site 289) with the assumption of a constant sedimentation rate. The FAD of *Orbulina suturalis* (N.8/N.9 boundary) was evaluated first and then used as a calibration point for the other biozone boundaries, even though this *Orbulina* datum was not constrained with radiometric ages (Fig. 1). The first occurrence (FO) of the genus *Orbulina* was found within the Neogene sequence in the Kii Peninsula, Southwest Japan. Felsic intrusive rocks in the sequence were dated to give an upper limit for the *Orbulina* datum (Ikebe et al., 1975; Shibata, 1978). The *Orbulina* datum is thought to be older than the K-Ar age of 14.5 ± 0.5 Ma, while the stratigraphic position of the FO of the genus *Orbulina* is located more than 700 m below the dated felsic rocks.

In contrast, the lower age limit of the *Orbulina* datum was defined in the Hokuriku district, on the Japan Sea side of central Japan. Dated samples of andesite lava are located 300–500 m below the extinction level of *Globigerinoides sicanus*. The FAD of *Gds. sicanus* defines the lowest limit of the zone N.8, so that the *Orbulina* datum (posterior to the FAD of *Gds. sicanus*) should be younger than that radiometric age (Tsuchi, 1983). Despite the large stratigraphic thicknesses between the FO of the genus *Orbulina* and each dated sample, Tsuchi et al. (1981) adopted a rounded number of 15.5 Ma as the age of the *Orbulina* datum in Japan.

This ambiguity in the age of the *Orbulina* datum has affected age estimations for other datum levels. Tsuchi et al. (1981) calibrated the age of each datum level based on a constant sedimentation rate using deep-sea core (Fig. 1). They utilized two calibration points of the FADs of *Pulleniatina* (5.8 Ma) estimated by Saito et al. (1975), and of *Orbulina* (15.5 Ma) evaluated as discussed above. The FAD of *Grt. peripheroacuta*, defining the N.9/N.10 boundary of planktonic foraminiferal zones, was recognized at level 460 m in the core, so that an age of 14.6 Ma was estimated as the N.9/N.10 boundary, assuming a constant sedimentation rate.

To avoid such ambiguous assumptions, as well as difficulty in estimations, it is more appropriate to determine the radiometric ages of tuff layers which are well controlled by the biostratigraphy. Ideally, each biozone boundary should be constrained with radiometric ages from both overlying and underlying tuff layers. Other measurements (e.g. magnetostratigraphy, chemostratigraphy) together with a chronostratigraphic investigation of the same section should confirm the magneto-biostratigraphic time scale.

3. Geologic Setting and Sampling

The Boso Peninsula is one of the best areas in Japan to study late Cenozoic stratigraphy, because thick marine sediments yielding calcareous microfossils from most horizons are intercalated with a large number of volcaniclastic layers. This sequence of fossiliferous Neogene and Pleistocene strata has only one stratigraphic break, named the Kurotaki Unconformity. Below the Kurotaki Unconformity, the thick marine sediments are sub-divided in ascending order into the following five formations: the Kanigawa, Kinone, Amatsu, Kiyosumi and Anno Formations. The lower half of this sequence is composed mainly of mudstones, while sandstones are dominant in the Kiyosumi Formation. The Anno Formation is characterized by rhythmic alternations of sandstone and mudstone (Figs. 2 and 3).

Magnetostratigraphy was first applied to this sequence by Kawai (1951), but only the present-day direction of the earth's magnetic field was recognized due to failure in removing a secondary component of the remanent magnetization. Nakagawa et al. (1969) established a reversal sequence for the detrital remanent magnetization, and Kimura (1974) supplemented and partly revised it. The latest revision of the magnetostratigraphy was made by Niitsuma (1976) applying thermal- and alternating-field demagnetization techniques. However only the last 6 m.y. history was established owing to magnetic instability (Nakagawa et al., 1977).

Detailed biostratigraphic studies (Oda, 1977; Honda, 1981) in conjunction with lithostratigraphic
investigations reveals the relation between many biostratigraphic events and key tuff layers as shown in Fig. 3. In contrast, the number of tuff layers in this sequence having radiometric ages is very small. Both the Amatsu and Anno Formations contain more than 100 tuff layers, and about 30 tuff beds are found intercalated in the Kiyosumi Formation, however only a few fission track ages were reported by Kasuya (1987, 1990). About one half of these tuffs are composed of scoria, which are not suitable for dating. Although the remainder are felsic tuffs, biotite and/or hornblende are rare. Thus, only about 10% of the total tuff layers contain minerals suitable for K-Ar age determination.

Some tuff layers in this sequence are well controlled by biostratigraphy. Among them, the Kn-3 Tuff is located fortuitously near the biozone boundary. The pumice tuff Kn-3 in the Kinone Formation is located about 10 m above the FO of Grt. peripheroacuta (Oda, 1977). Grt. peripheroacuta is one of the most important key species of planktonic foraminifera, because its FAD defines the N.9/N.10 boundary of planktonic foraminiferal zones (Blow, 1969). Based on the stratigraphic interval between the Kn-3 Tuff and the FO of the Grt. peripheroacuta (approximately 10 m), the time lag of these two horizons may be negligible, because the mean sediment accumulation rates were 300 m/m.y. or more for the Kinone Formation, and 100 m/m.y. for the Amatsu Formation. The duration for the accumulation of the 10 m-thick sediments is 0.1 m/m.y. or less, which is shorter than the error obtained for the radiometric age. Consequently, it can be regarded that the radiometric age of the Kn-3 Tuff represents the age of the N.9/N.10 boundary in the Boso sequence in central Japan.

We collected more than 30 kg of the Kn-3 Tuff from the lower part in the Meigawa section (Fig. 4). The Kn-3 Tuff consists of massive pumice tuff and laminated pumiceous tuff. As it contains neither biotite nor hornblende, we attempted to date it by the fission track method using zircon. The refractive indexes of volcanic glass and plagioclase from the Kn-3 Tuff are listed in Table 1.

4. Experimental Procedure

Approximately 15 kg of tuff sample was crushed and zircon grains were concentrated by sieving, panning, magnetic and heavy liquid separating techniques. More than 1000 zircon grains were obtained, of which 30% of the grains were regarded as essential. Full details of the careful recognition of essential and nonessential zircon grains will be outlined.
Zircon samples were analyzed using the external detector method (ED2 method: Gleadow, 1981). Zircon grains were mounted onto a Toyoflon PFA sheet (Danhara et al., 1993), with the crystal surfaces exposed. The zircon mount was etched in a eutectic mixture of NaOH and KOH (Gleadow et al., 1976) at 225°C for 28 hours. Low-uranium muscovite was attached to the zircon mounts as an external detector for the induced tracks. Neutron irradiation was carried out on the Rotary Specimen Rack in the TRIGA Mark II reactor at St. Paul's University. Thermal neutron fluences were monitored by using a NBS-SRM612 dosimeter glass attached with muscovite detectors. In order to detect neutron flux gradients, the piled mount was sandwiched between two sets of the dosimeter glass. After irradiation, the muscovite detectors were etched in 46% HF at 20°C for 24 minutes for the zircon mounts, and 44 minutes for the dosimeter glasses. Track counting was carried out using an overall magnification of 1000×. Zircon grains with well etched and isotropically distributed tracks were selected for the counting. See Danhara et al. (1991) for further details.
Fig. 4. Detailed geological route map and stratigraphy of the Meigawa section, with the location of dated samples.
Table 1. Refractive index of volcanic glass and of plagioclase in the Kn-3 Tuff.

| Minerals      | Min. | Max. | Mean | No. | Mode       | Commentary         |
|---------------|------|------|------|-----|------------|--------------------|
| Glass         | 1.502| 1.508| 1.505| 30  | 1.504 < Nd < 1.507 | Tube type          |
| Plagioclase   | 1.552| 1.558| 1.554| 30  | 1.552 < Nd < 1.555 | Andesine-labradorite|

Fig. 5. Relationship between previously reported estimated ages of the N.9/N.10 boundary of planktonic foraminiferal zones and the fission track age of the Kn-3 Tuff, which is directly correlated to the age of the boundary.

A fission track age $T$ is calculated using the following equation (Price and Walker, 1963; Naeser, 1967):

$$ T = \left(\frac{1}{\lambda_d}\right) \ln \left[1 + \lambda_d \zeta g (\rho_s/\rho_i) \rho_d \right] $$

where $\lambda_d =$ total decay rate for $^{238}$U, $(\rho_s/\rho_i) =$ spontaneous/induced track density ratio in the sample, $\rho_d =$ induced track density for a dosimeter glass, and $g =$ geometry factor. The age calibration was made by the zeta ($\zeta$) approach (Hurford and Green, 1983). The zeta value of $372 \pm 5$ and the geometry factor of 1 $(2\pi/2\pi)$ were used for the ED2 method (Danhara et al., 1991). Errors were calculated using the “conventional analysis” of Green (1981).
Table 2. Fission track data of zircons from the Kn-3 tuff in the late Cenozoic sedimentary sequence in the Boso Peninsula, central Japan. Kn-3 (dt.) is the result of the detrital (non-essential) zircons for comparison.

| Sample code | No. of crystals | Spontaneous | Induced | P(χ²) | Dosimeter | r | U-content | Age (±1σ) |
|-------------|-----------------|-------------|---------|-------|-----------|---|-----------|-----------|
|             |                 | ρ₁ (10⁶ cm⁻²) | N₁     | ρᵢ (10⁶ cm⁻²) | Nᵢ | ρ₀ (10⁶ cm⁻²) | N₀ |        | (ppm) | (Ma) |
| Kn-3 (1)    | 29              | 1.94        | (634)   | 3.42  | (1115)    | 51 | 7.10      | (1093)   | 0.816 | 390  | 15.0 ± 0.9 |
| Kn-3 (2)    | 30              | 1.50        | (622)   | 2.83  | (1171)    | 66 | 7.77      | (1198)   | 0.943 | 290  | 15.3 ± 0.9 |
| Kn-3 (3)    | 33              | 1.74        | (646)   | 3.42  | (1267)    | 97 | 7.77      | (1198)   | 0.906 | 350  | 14.7 ± 0.9 |
| Kn-3 (dt.)  | 11              | 11.4        | (1025)  | 3.26  | (292)     | 5  | 7.77      | (1198)   | 0.147 | 340  | 101 ± 7    |

ρ and N are the density and the total number of fission tracks counted, respectively.
Analyses were made by the external detector method using geometry factor of 1 for 2π/2π (ED2).
Ages were calculated using a dosimeter glass SRM612 and age calibration factor ζ (ED2) = 372 ± 5 (Danhara et al., 1991).
P(χ²) is the probability of obtaining the χ²-value for ν degrees of freedom (where ν = number of crystals − 1).
r is the correlation coefficient between ρ₀ and ρᵢ.
Samples were irradiated using TRIGA MARK II nuclear reactor of St. Paul's University (Rikkyo Daigaku), Japan.
After etching the spontaneous tracks in zircon, two groups of zircons were recognized: (1) lower spontaneous track density \( (\rho_s) \) of \( 1 - 2 \times 10^6 \) tr/cm\(^2\), and (2) higher track density of \( 1 \times 10^7 \) tr/cm\(^2\) or greater. Three zircon mounts were prepared and irradiated for two different runs in order to confirm the reproducibility of the fission track age in the sample. About 30 zircon grains with lower spontaneous track density were examined on each mount. For comparison, fission track ages of the zircon with higher track density were also measured.

5. Result and Discussion

The fission track data for the Kn-3 Tuff is shown in Table 2. Fission track ages from three sets of the low spontaneous track density \( (\rho_s) \) zircons agree with one another within the acceptable errors. All three measurements pass the \( \chi^2 \)-test (Galbraith, 1981) at the 5% level, which strongly suggests that most of the grains belong to a single age population and do not contain any significant detrital component. The weighted-mean age has been calculated to be \( 15.0 \pm 0.5 \) Ma for this zircon group. For the high \( \rho_s \) zircon group, a fission track age was determined to be \( 101 \pm 7 \) Ma, significantly older than the low-\( \rho_s \) zircon group. Consequently, we found that there are at least two age components in the zircons from the Kn-3 Tuff, and concluded that the younger group \( (15.0 \pm 0.5 \) Ma; \( 1 \sigma \) error) represents the formation age of the Kn-3 Tuff.

The fission track age of the Kn-3 Tuff gives an age to the N.9/N.10 boundary of planktonic foraminiferal zones in the Boso sequence, as the FO of Grt. peripheroacuta is recognized near the dated tuff horizon.

Figure 5 shows the relationship between the fission track age of the Kn-3 Tuff and previously reported geochronometric ages for the N.9/N.10 boundary, estimated from each time scale. It is clear that most of the recent estimates for the N.9/N.10 boundary are concordant with the fission track age of the Kn-3 Tuff which precisely indicates the age of the boundary.

The N.9/N.10 foraminiferal boundary in the sedimentary sequence in the Boso area, central Japan, is correlated to a radiometric age by this work. Magnetostratigraphy along this sequence however still has to be established. If we assume the synchronism of the FAD of Grt. peripheroacuta between latitudinal areas, the fission track age of the Kn-3 Tuff may suggest that the numerical age of the N.9/N.10 boundary of each time scale should be equal to the age of the Kn-3 \( (15.0 \pm 0.5 \) Ma; \( 1 \sigma \) error).

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