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Effect of flash thermal annealing by pulsed current on rotational anisotropy in exchange-biased NiFe/FeMn film*

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In this paper, Ta/[NiFe(15 nm)/FeMn(10 nm)]/Ta exchange-biased bilayers are fabricated by magnetron sputtering, and their static and dynamic magnetic properties before and after rapid annealing treatment with pulsed current are characterized by using a vibrating sample magnetometer (VSM) and a vector network analyzer (VNA), respectively. The exchange bias field H_e and static anisotropy field H_k^{sta} decrease from 118.45 Oe (1 Oe = 79.5775 A·m^{-1}) and 126.84 Oe at 0 V to 94.75 Oe and 102.31 Oe at 90 V, respectively, with increasing capacitor voltage, which supplies pulsed current to heat the sample. The effect of flash thermal annealing by pulsed current on the rotational anisotropy (H_{rot}), the difference value between static and dynamic magnetic anisotropy, is investigated particularly. The highest H_{rot} is obtained in the sample annealing with 45-V capacitor (3300 μF) voltage. According to the anisotropic magnetoresistance measurements, it can be explained by the fact that the temperature of the sample is around the blocking temperature of the exchange bias system (T_b) at 45 V, the critical temperature where the formation of more unstable antiferromagnetic grains occurs.

Keywords: rapid annealing, sputtering, pulsed current, rotational anisotropy

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1. Introduction

It is pressing to develop a series of soft magnetic films for high-performance inductors, noise filters, magnetic recording heads, and microwave device, with the increasing development of information technology and mobile communication technology.[1] The ferromagnetic resonance frequency, f_r, an important parameter of high frequency devices, can be adjusted to a higher value by the exchange bias effect between ferromagnetic (FM) layer and antiferromagnetic (AFM) layer, and by introducing an additional unidirectional anisotropy into the ferromagnetic layer.[2,3] Generally, the unidirectional anisotropy and ferromagnetic resonance frequency increase with increasing AFM layer thickness within limits, beyond which there will be no dependency between them. However, according to our previous publication,[4] the ferromagnetic resonance frequency of the FM/AFM film with a thin AFM thickness can be higher than that with a thicker AFM thickness. That is, in an FM/AFM exchanged bias system, the dynamic anisotropy is much higher than static anisotropy.[5] Rotatable anisotropic field, which may be caused by the different types of AFM grains, was introduced to explain this phenomenon by Stiles and McMichael.[6–8] The frozen spin grains, with large uniaxial anisotropic energy (K_{AFM}), responsible for static anisotropy can be obtained by static measurement, such as a vibrating sample magnetometer (VSM),[9] while the rotatable spin grain, with weaker K_{AFM} is responsible for rotatable anisotropy,[10] which cannot be obtained by static measurement but can be obtained by dynamic measurement. So the difference between the values of static anisotropy and dynamic anisotropy field (H_k^{dyn}) is a rotatable anisotropy field. In some works in the literature[7,8] it was proposed that the frozen spin grains be in a magnetically stable state, while the rotatable spin grains be in a magnetically unstable state.

Pulsed current has been used in the fields of material science and technology, based on its peculiarity in rapid heating and cooling, to obtain special performance, such as electroplastic effect,[11] electromigration effect,[12] rapid recrystallization[13,14] etc. Fe–Ni–Si–B metallic glasses were annealed and crystallized using short current pulses by Zaluski *et al.*[15] The sample initially held in liquid nitrogen and rapidly heated with a pulsed current (fast heating and cooling) remains amorphous and shows improved magnetic properties, including lower coercive field and higher induction at saturation, without loss of ductility.

In this paper, the pulsed current peculiarity in rapidly heating and cooling is used for tailoring the rotatable anisotropy by affecting the magnetic stability of AFM grains in NiFe/FeMn exchange biased films. By altering pulsed current, different rotatable anisotropies in the same

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batch of samples are obtained.

2. Experiment

Samples with the structure of Ta (5 nm)/NiFe (15 nm) /FeMn (10 nm)/Ta (5 nm) were fabricated onto Si (100)/SiO_2 (500 nm) substrates by using direct current magnetron sputtering at ambient temperature. The base pressure was about 2×10^{-5} Pa prior to deposition. The Ar pressure was kept at 0.4 Pa. A 5-nm-thick Ta buffer layer was introduced to promote the growth of AF face-centered-cubic (FCC) FeMn with preferred (111) orientation and the top layer was coated to protect them from being oxidized. All the layers were made from alloy targets, the purities of targets were 99.99% (Ta), 99.99% ($Ni_{80}Fe_{20}$), and 99.95% ($Fe_{50}Mn_{50}$), respectively. During deposition, a constant magnetic field of 200 Oe was applied parallel to the film plane to induce the exchange bias.

The schematic diagram of rapid annealing experiment by pulsed current is shown in Fig. 1. The sample with a 200-nm-thick copper electrode, which ensures the homogeneity of current flowing through samples, was held in a vacuum chamber, and a constant magnetic field of 200 Oe was applied parallel to the film plane along the exchange bias direction. Firstly, with switch 2 turned off, while switch 1 turned on, the capacitor was charged to a certain voltage (0, 45, 65 or 90 V) through controlling the power source voltage. After the capacitor was fully charged, i.e., the capacitor voltage reached the power source voltage, which took less than 0.2 s, we turned off switch 1 then turned on switch 2, the capacitor discharged its 99% energy in 0.16 s to the sample, rendering its rapid heating. The conduction of heat between film and substrate caused it to rapidly cool. Each sample was treated only once.

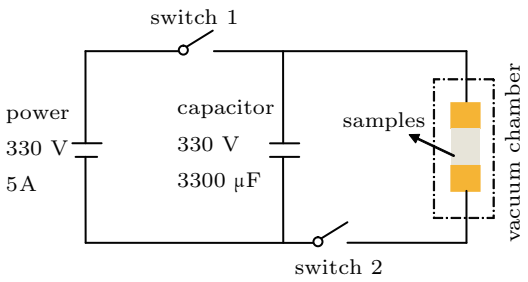


Fig. 1. (color online) Schematic diagram of rapidly annealing experiment by pulsed current.

The static magnetic properties of the samples before and after being treated with pulsed current were measured by vibrating sample magnetometer (BKT-4500Z). The microwave permeability measurements of the films were carried out by a vector network analyzer (E8363B) using the shorted micro strip transmission-line perturbation method.[4,10]

The anisotropic magnetoresistance (AMR) values of samples before and after being treated with pulsed current (different from the aforementioned process, a constant magnetic field of 200 Oe was applied parallel to the film plane and oppositely to the exchange bias direction) were measured by four-point probe method to pinpoint the critical voltage of the capacitor, at which the temperature of the sample is over the blocking temperature of the exchange bias system. With the increase of capacitor voltage, if the exchange bias direction just reversed, the capacitor voltage was considered to be the critical voltage to heat the film to a temperature over T_b. The schematic of our measurement is shown in Fig. 2, indicating that the exchange bias (H_e) direction is parallel to the current (I) direction and tilts 30° (φ_H) with respect to the direction of external magnetic field (H_{ext}).

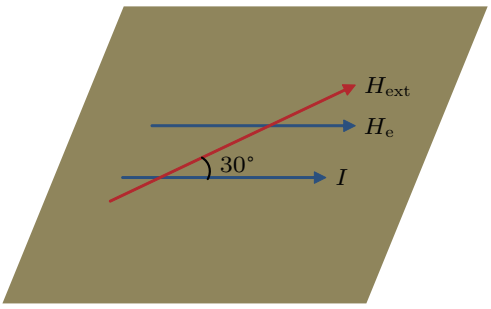


Fig. 2. (color online) Schematic diagram of measurement of AMR.

Besides, for convenience, H_e^{MR} was defined as the applied magnetic field, and it was scanned from +200 Oe to −200 Oe then back to 200 Oe, corresponding to the right minimum of resistance in AMR measurement.

3. Results and discussion

Figure 3 shows the hysteresis loops of bilayers treated with 0, 45, 65, and 90 V capacitor voltages, respectively, and measured at room temperature. The easy axis (EA) direction is defined as the direction of the field applied during deposition while the hard axis (HA) is in the direction normal to the easy axis but still in the plane of the sample.[16,17] As shown in Fig. 3, the shapes of the hysteresis loops have little changes, which indicates that the structures of the samples are not destroyed by the pulsed current. The H_e and H_k^{sta}, both extracted from hysteresis loops presented in Fig. 3, of the raw sample are 118.45 Oe and 126.84 Oe. The H_k^{sta} is extracted from the slope of rotational-like magnetization curve on the HA, which is the sum of the intrinsic uniaxial anisotropy field (H_k^{int}) of the FM layer and the exchange bias field (H_e).[8,18] When the sample was treated with pulsed current, the H_e and H_k^{sta} decrease with increasing capacitor voltage as seen in Fig. 4(a). We notice that the difference values between H_k^{sta} and H_e, i.e., the values of H_k^{int}, in Figs. 3(a)–3(d), are all about 8 Oe, which is due to the FM (NiFe) layers being the same thickness (15 nm).

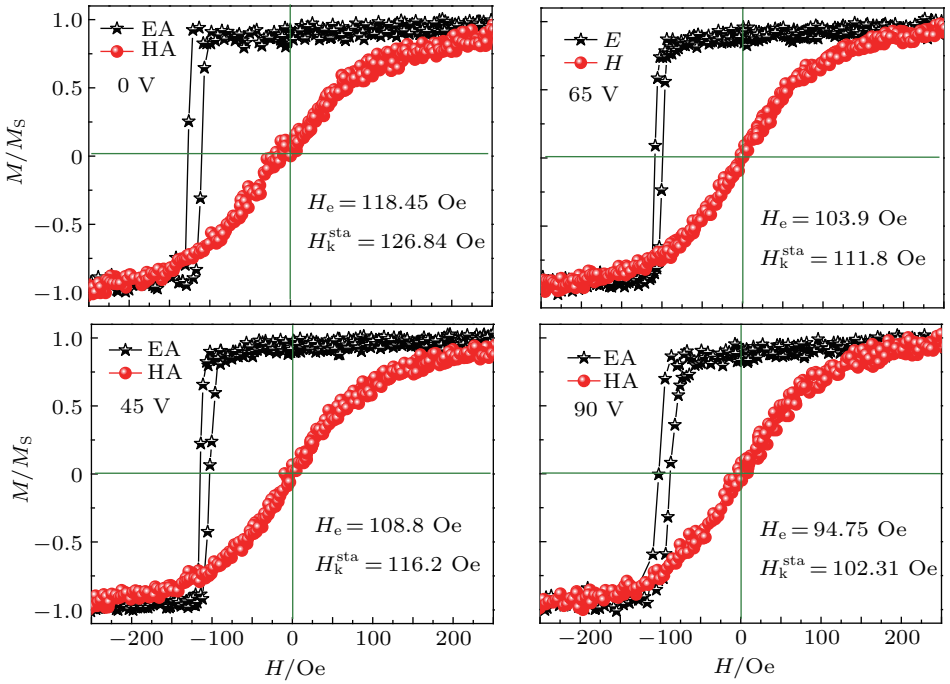


Fig. 3. (color online) Hysteresis loops of NiFe(15 nm)/FeMn (10 nm) bilayers before and after being treated with pulsed current.

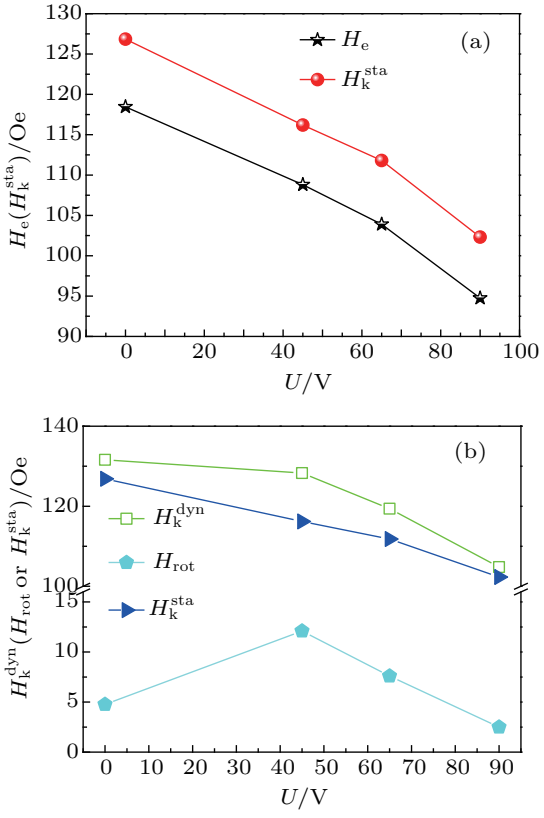


Fig. 4. (color online) (a) Variations of exchange bias field (H_e) and static anisotropy field (H_k^{sta}) with capacitor voltage. (b) Comparison among dynamic magnetic anisotropy (H_k^{dyn}), H_k^{sta} and rotatable anisotropy (H_{rot}) as a function of capacitor voltage.

In order to investigate the changes in dynamic magnetic properties of the samples, a shorted microstrip transmission-line perturbation method[4,10] is employed to characterize their dynamic magnetic properties. The permeability spectra with real μ' and imaginary μ for samples treated with different capacitor voltages were shown in Fig. 5 in a frequency range of 2 GHz∼ 6 GHz. It can be observed that the peak of the imaginary permeability is slightly shifted to the lower frequency range due to the decrease of H_e. For the purpose of quantitatively analyzing the dynamic magnetization versus pulsed current, the famous Landau–Lifshitz–Gilbert (LLG) equation[19,20] is used to fit the experimental permeability spectra. The fitting method and corresponding formulas have been displayed in our previous publications.[4] In the LLG equation, M and H represent the magnetic moment and magnetic field, respectively, γ denotes the gyromagnetic ratio, and α_{eff} refers to the dimensionless effective damping coefficient.

The capacitor voltage dependent exchange bias field (H_e) and static anisotropy field (H_k^{sta}) are shown in Fig. 4(a). The H_e and H_k^{sta} vary about 20% when the capacitor voltage offering pulsed current changes from 0 V to 90 V. From Fig. 4(b), it can be seen that there is a difference between static and dynamic magnetic anisotropy field. It can be explained by the contribution of rotatable anisotropy ($H_{rot} = H_k^{dyn} - H_k^{sta}$).[5,18] This phenomenon has been observed in other exchange biased systems[3,5,19] and can be explained as follows. In static magnetic measurement such as VSM, rotational anisotropy cannot be measured directly because the unstable AF grain magnetization, the origin of rotational anisotropy, has enough time to rotate and follow the magnetization on this time scale. However, in dynamic measurement the small excited radio frequency magnetic field, which changes directions at microwave frequency, is not large enough to make the magnetization of an unstable AF grain reverse. Hence, dynamic measurement can detect the existence of the rotational magnetic anisotropy

while the static measurement cannot.[7,8,18]

The resonance frequency (f_r), frequency linewidth (Δf), and effective damping coefficient (α) are also shown in Fig. 5. The method of fitting α and formulas for calculating Δf have been displayed in our previous publication.[4] Frequency linewidth and effective damping coefficient do not change much with capacitor voltage, implying that the frequency linewidth behavior is mainly dependent on the effective damping coefficient.[4,18]

As seen in Fig. 4(b), H_k^{sta}, and H_k^{dyn} both decrease with increasing capacitor voltage, but their difference at 45 V has a maximum, that is, H_{rot} reaches its peak value on this condition. When capacitor voltage surpasses 45 V H_{rot} diminishes with increasing annealing voltage (temperature). According to the aforementioned H_{rot} derived from the unstable grains in AFM layer, whether annealing at 45 V capacitor voltage can cause more rotatable spin AFM grains to arise, which arouses our interest.

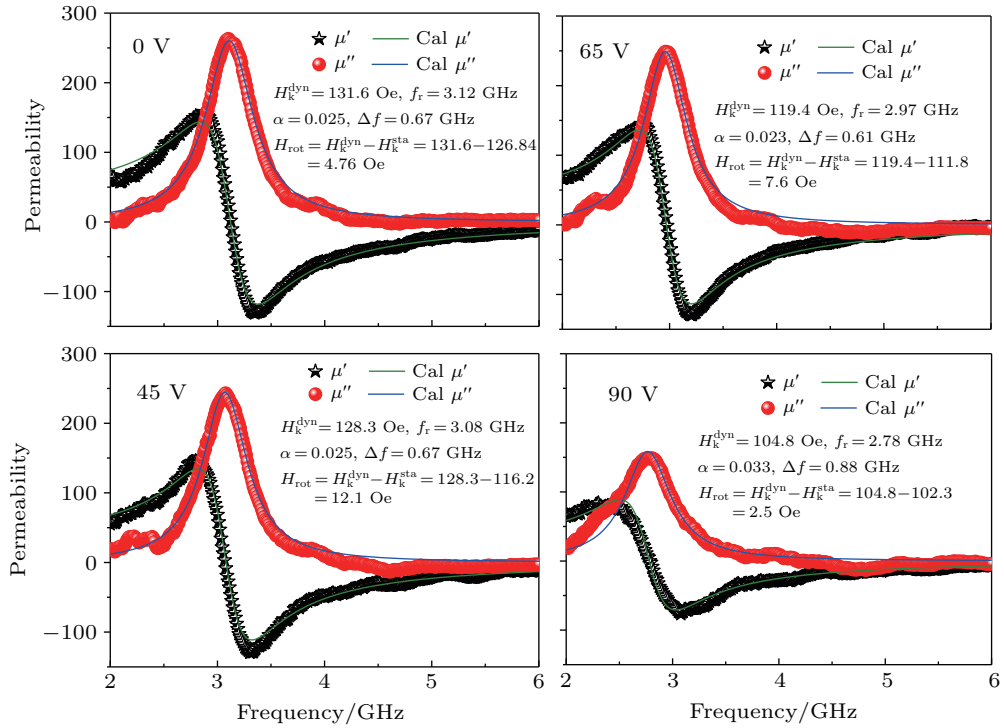


Fig. 5. (color online) Real and imaginary permeability spectra of NiFe(15 nm)/FeMn(10 nm) bilayers treated with different pulses currents. The lines are fitting curves based on the LLG equation.

The anisotropic magnetoresistance measurement is employed to investigate the reason for the variation trend of H_{rot} of [NiFe(15 nm)/FeMn(10 nm)] treated with different pulsed current.[21–23] As revealed in Fig. 6, H_e^{MR} is a characteristic value in the AMR spectrum of exchange biased film, and relates to H_e and the angle (φ_H) between external magnetic field (H_{ext}) direction and the exchange bias (H_e) direction. When φ_H is fixed at 30°, H_e^{MR} is directly proportional to the H_e value of the sample.[23,24] So we determine the new exchange bias direction after annealing in the external magnetic field (H_{ext}) reversed to exchange bias direction by H_e^{MR}. It should be stated here that the exchange bias (H_e) direction depicted in Fig. 2 is that of the raw sample, although for the samples annealed by 45 V or 50 V their exchange bias directions have reversed, which is the reason for the opposite shapes of the curves in Figs. 6(a) and 6(b) compared with Figs. 6(c) and 6(d). The H_e^{MR} initial value of the sample is 89.3 Oe. When it is treated with pulsed current supplied by 40 V charged capacitor in a constant magnetic field of 200 Oe, H_e^{MR} decreases to 49.0 Oe but the exchange bias direction does not reverse, which indicates that the stable grains in the AFM layer diminishes under the effect of reversed magnetic field and the temperature of the sample may be close to its T_b value.[4,8,18] Here, the external magnetic field is applied parallel to the film plane and oppositely to the exchange bias direction. With the capacitor voltage increased to 45 V, the exchange bias field reverses but its value does not reach to 89.3 Oe as shown in Fig. 6(c), the value of raw sample, which illustrates a higher temperature around T_b is generated in exchange biased film and most of AFM and FM magnetizations switch to the opposite direction.[25,26] The exchange bias field almost completely reverses its direction and its value returns to 88.2 Oe when the capacitor voltage further increases to 50 V as shown in Fig. 6(d). In dynamic magnetic measurement the highest H_{rot} is obtained with a 45-V capacitor voltage as revealed in Fig. 4(b), which can be ascribed to the critical temperature at 45 V annealing according to the results of AMR tests.

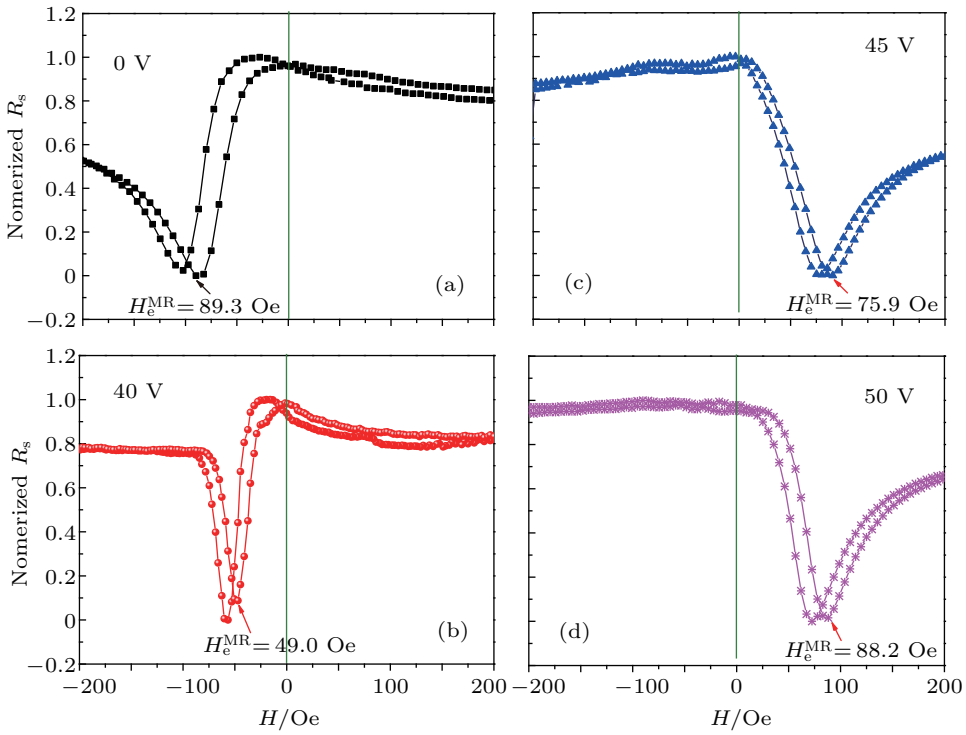


Fig. 6. (color online) AMRs of NiFe(15 nm)/FeMn(10 nm) bilayers treated with different pulsed currents.

In order to further discuss the variation of H_{rot} with annealing voltage a schematic diagram of the spin configuration in bilayers treated with pulsed current is shown in Fig. 7. The AFM layer in a coupled AFM/FM system is characterized by both frozen and the rotatable spin grains. As previously mentioned the unstably rotatable AFM grains determine the H_{rot} and always exist in AFM/FM exchange biased film as shown in Fig. 7(a). During 45-V annealing the film temperature increases rapidly to approximate T_b, where the AFM spin becomes disordered. As the temperature rapidly lowers to below T_b part of the AFM grains keep the state at high temperature, which brings about the increased unstable and rotatable AFM grains, and then the increased H_{rot} as displayed in Fig. 7(b).[7]

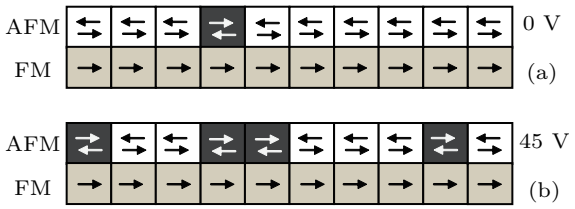


Fig. 7. (color online) Schematic diagram of the spin configuration in NiFe(15 nm)/FeMn(10 nm) bilayers treated with 45-V anealing.

4. Conclusions

Static and dynamic anisotropy in NiFe(15 nm)/FeMn(10 nm) exchange-biased bilayers before and after being rapidly annealed with pulsed current are investigated by using a vibrating sample magnetometer and a vector network analyzer. The exchange bias field H_e and static anisotropy field H_k^{sta} decrease with increasing capacitor voltage. The discrepancy between static and dynamic anisotropy (H_{rot}) reaches its maximum by annealing with 45-V capacitor (3300 μF) voltage, which can be explained by the fact that the temperature of the sample is around the T_b of the exchange bias system. Under this critical temperature more unstable AFM grains are formed, which leads to the highest H_{rot}.

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