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STROBOSCOPIC TECHNIQUE FOR DYNAMIC OBSERVATION OF FERROMAGNETIC DOMAINS AT LOW FREQUENCIES IN SCANNING ELECTRON MICROSCOPY

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Abstract

A new stroboscopic technique for the observation of periodic phenomena at low frequencies (10 - 100 Hz) in scanning electron microscopy (SEM) is developed in the present study. This new technique is based on the line-sampling stroboscopy instead of the point-sampling stroboscopy which has been commonly used for the dynamic observation at high frequencies (> 1 kHz). In this stroboscopy, a multi-frame display with the individual stroboscopic image at different phase can be easily introduced with no reduction of the detection efficiency.

The main application of the present stroboscopy at low frequencies is the dynamic observation of magnetic domains. In this case, high voltage scanning electron microscopy is very effective to increase the magnetic contrast as pointed out by several workers. According to this, dynamic observation of Fe-Si 3% single crystals under a drive field of line frequency (60 Hz) using a 200 kV high voltage SEM (JSEM-200, JEOL Co. Ltd.) is achieved in this paper, as a typical application of this new stroboscopy. From these dynamic observation, it is concluded that this new technique is a very useful tool to analyze the dynamic property of magnetic materials in practical use.

Dedicated to the Professor Emeritus E. Sugata on occasion of his 77th birthday.

KEY WORDS: Strobosopic scanning electron microscopy, Line-sampling multi-frame stroboscopy, Type-II magnetic contrast, Silicon-steel

Introduction

Dynamic observation of periodic phenomena using scanning electron microscopy (SEM) has been attempted by Plows and Nixon (1968) using a point-sampling stroboscopic technique to observe the dynamic response of microcircuit voltage distribution, and also MacDonald et al (1969) to observe moving high field domains in a bulk effect oscillator. Now, the time resolution achieved with this stroboscopy is about 1.5 ps (Hosokawa et al 1978) by using primary beam strobing.

The lower limitation with the frequencies of the repetition in the point-sampling stroboscopy is practically limited by the scanning time. In most scanning electron microscopes, the maximum scanning time to get one frame is up to 1000 seconds. And if 1000 x 1000 pixels are required for one stroboscopic image, the minimum sampling rate (pixels/scanning time) is the order of 1000 pixels/second. This value is obviously identical with the repetition frequency in the point-sampling stroboscopy. Hence it is concluded that the practical lower limit of the repetition frequency is about 1 kHz.

On the other hand, the use of the scanning electron microscopy for the observation of magnetic domains through a backscattered electron image (type-II magnetic contrast) has been studied (Philibert and Tixier 1969, Fathers et al 1973, Newbury et al 1973, Ikuta and Shimizu 1974) and confirmed to be very useful for the static observation of the magnetic domains in ferromagnetic materials in practical use.

For the simple evaluation of the dynamic response of magnetic domains, some workers (Yamamoto and Tsuno 1976, Livingston and Morris 1981, Washko et al 1982) have already reported synchronous drive methods in which the field driving is synchronized with the SEM horizontal sweeping. These methods are, however, not based on the true stroboscopic technique and restricted for only the observation with the one-dimensional response of the magnetic domains.

A first application of the true point-sampling stroboscopy for ferromagnetic samples has been reported by the author (Ikuta and Shimizu 1976), where an electronic gating of the video signals has been used for the observation of ferromagnetic domains in silicon-steel sample at audio frequency (1 kHz) without any reconstruction of the electron optical system.

It is, however, very clear that the most important frequency region for the stroboscopic observation of the ferromagnetic domains is the lower region such as line frequencies (50 - 60 Hz), since many ferromagnetic materials in power system are used in this frequency region. Regretfully as mentioned above, the direct application of the point-sampling stroboscopy at the line frequency is very difficult at this time.

This paper describes an attempt to extend the stroboscopic technique for the dynamic observation of magnetic domains at the lower frequency region (based on a newly developed line-sampling multi-frame stroboscopy (Shimizu and Ikuta 1984)) and the usage of high voltage scanning electron microscopy to obtain clear stroboscopic images of magnetic domains. Photographs of the dynamic observation of magnetic domains in silicon-steel samples, obtained by a 200 kV high voltage scanning electron microscope (JSEM-200, JEOL Co. Ltd.) are presented to demonstrate this new stroboscopy.

Line-sampling multi-frame stroboscopy

As described in the previous section, Shimizu and the author have proposed a new stroboscopic observation technique (Shimizu and Ikuta 1984) which enables a dynamic observation of the periodic phenomena in the low frequency regions, including the commercially used power frequencies (50 - 60 Hz). In Fig. 1(a), schematic diagrams of the conventional point-sampling stroboscopy are illustrated. In the conventional stroboscopy, one pixel of the stroboscopic image just corresponds to one sampling phase in the periodic driving. On the other hand, in the proposed new stroboscopy, one scan line corresponds to the sampling phase as shown in Fig. 1(b). Hereafter this technique is referred to as line-sampling stroboscopy. In this line-sampling stroboscopy, faster scan speed is desirable for the horizontal scanning of the SEM because of the requirement that the phase of the field drive remains sensibly constant over the time interval for the one horizontal scan line.

A main advantage of this line-sampling stroboscopy is its faster image construction time due to the multi-point data acquisition during the interval of one line-sampling within one cycle of the driving. For example, a line-sampling stroboscopic observation at 60 Hz driving takes about 10 - 20 seconds of the image reconstruction time. This time is faster than conventional stroboscopy for about 1000 times (corresponding to the number of pixels on the horizontal scan line).

Another advantage of the present stroboscopy is that it allows the observation of several stroboscopic images for different phase values at the same time. This technique is illustrated in Fig. 2. A multiple line-sampling at different phase value in one cycle of the field drive can be applied to the present line-sampling stroboscopy. Resulting multiplexed video signals are spatially redistributed on the monitor CRT by summing up appropriate translation signals to the vertical and horizontal deflection signals. Namely the images with different phase value may be displayed on the CRT by dividing the scan area into several

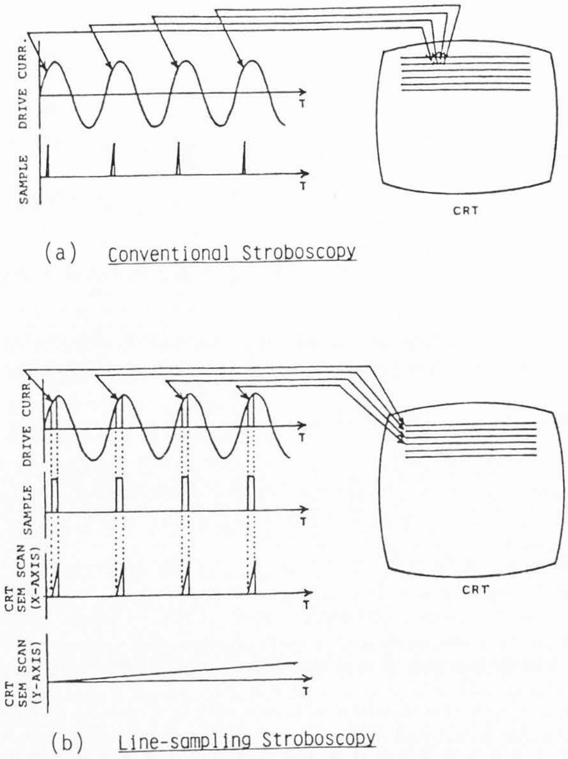


Fig. 1. Schematic diagrams of (a) conventional point-sampling stroboscopy and (b) line-sampling stroboscopy in the scanning electron microscope.

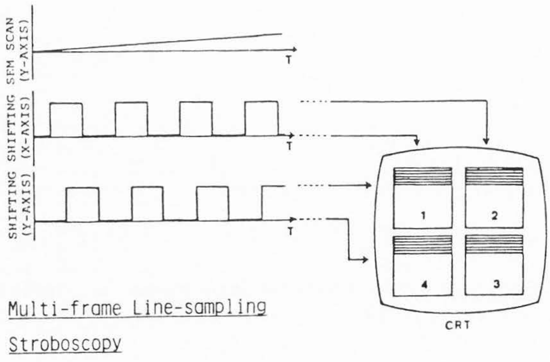


Fig. 2. Schematic diagrams of the multi-frame display technique combined to line-sampling stroboscopy shown in Fig. 1(b).

frames of different phase values as shown in this figure (this technique is referred to as multi-frame stroboscopic display). In addition to the fast image acquisition, this technique also provides an easy way for the operator to compare each stroboscopic image of different phase value at once.

System description and operation

At present, a primary beam strobing method has been utilized by most workers to realize a fast stroboscopic operation in scanning electron microscopy, as mentioned in the introduction of this paper. However, there is an alterative method to do this without any reconstruction of the electron optical system if the repetition of the stroboscopic observation is not so high. The author and Shimizu (Ikuta and Shimizu 1974) have reported a video signal gating method to observe the dynamic properties of magnetic domains in a Fe-Si sample under 1 kHz sinusoidal drive field.

In the present stroboscopy, the line-sampling is achieved by using the video signal gating of appropriate duration synchronized with the drive waveform. The gating must be also synchronized with the horizontal scanning, while no synchronization is required with the usual point-sampling stroboscopy.

While the beam strobing is not required in this system, a deflection coil must be replaced by a faster one to achieve the fast horizontal scanning at the duration of the line-sampling, if the scanning electron microscope used has only a slow beam scanning system. A fast detector (scintillation detector or fast silicon solid detector of about 1 μs temporal resolution) of backscattered electrons and wide-band video chains (several 100 kHz) are also required.

Fortunately such requirements have been already realized in the TV frame rate scanning electron microscopy (well known as TV-scan) which response is faster than that of the present line-sampling stroboscopy by about one order of magnitude.

As described above, the video signal gating must be synchronized to the sample driving and the horizontal beam sweeping in the line-sampling stroboscopy. In the present line-sampling stroboscopic apparatus, the gate signal, the horizontal sweep signal and the sinusoidal drive signal are all generated by using phase locked loops (PLLs) from a master clock signal of an internal clock generator to synchronize these signals with each other. In Fig. 3, schematic block diagrams of the present line-sampling stroboscopic apparatus are shown.

Two PLLs are used as synchronized generators of the horizontal scan signal (triangle waveform) and the sample drive signal (sinusiodal waveform). An additional PLL is also used to generate a sinusoidal signal whose phase difference from the drive is 90 degrees. This sinusoidal signal is used for the induction noise compensation with combination of the main drive signal. On the other hand, the vertical scan signal is not required to synchronize with these signals. An independent (not synchronized) scan generator is used for vertical scanning. In the multi-frame stroboscopy, appropriate deflection signals of a monitor CRT to display each stroboscopic image of different phase value simultaneously are generated using a PROM. This PROM is also used as a generator of blanking signals of the monitor CRT. In the present apparatus, 1- , 4- and 8- frames mode operations are possible.

In the first report of the dynamic observation with the magnetic domains at line drive frequency (Shimizu and Ikuta 1984), the instrument used was a commercial electron microprobe JAX-3 of JEOL Co. Ltd. . The observation was performed under the condition of 45 keV accelerating voltage. As many authors have demonstrated, the usage of a high voltage scanning electron microscopy improves the type-II magnetic contrast drastically (Newbury et al 1973, Shimizu et al 1974, Yamamoto et al 1975, Shimizu et al 1976 and Kinoshita et al 1976).Therefore the 200 kV high voltage scanning electron microscope has been used in the present study.

The improvement of the magnetic contrast in the 200 kV SEM is about one order of magnitude compared with the 45 kV acceleration. This enables high quality image of magnetic domains even in the stroboscopic observation.

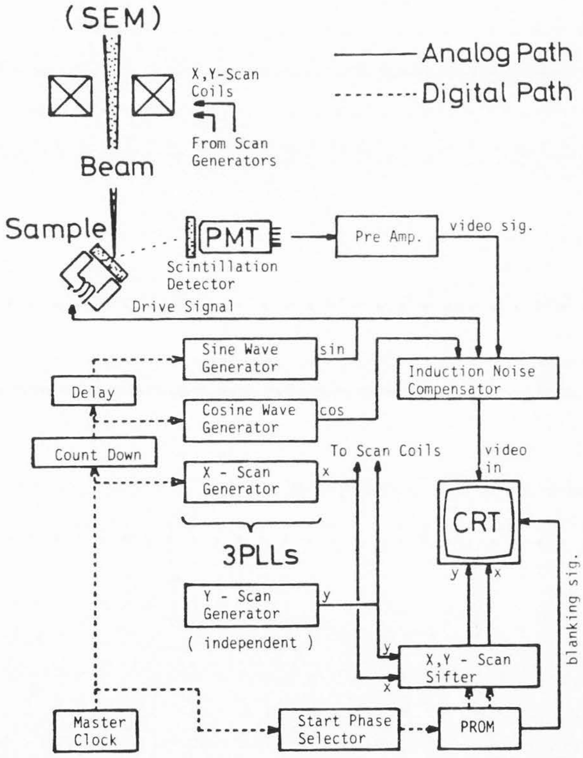


Fig. 3. Schematic block diagrams of the line-sampling multi-frame stroboscopic apparatus for the observation of the dynamic response of magnetic domains under the drive field of line frequency(50 - 60 Hz).

Experimental results and discussions

In the present experiment, a 200 keV operation of SEM (JSEM-200, JEOL Co.Ltd.) has been used to obtain good magnetic images as described above.

The samples used have been mounted on the open end of a truncated frame-shaped silicon-steel core wound around a field drive coil, and this assembly is fixed in the sample stage with the angle of incidence of 45 degrees to the electron beam. A scintillation detector with take-off angle of about 60 degrees from the sample surface is used as the detector of backscattered electrons which contain the information of the magnetic contrast.

In the following, the intensity of drive field will be denoted by using a drive current value (AT unit). True values of the drive field can be obtained from a relation 1 AT = 25 A/m .

A stroboscopic observation of magnetic domains in a Fe-Si 3wt% single crystal platelet of 0.3 mm thickness with orientation (100)[001] is shown in Fig. 4. Fig. 4(a) indicates a dynamic response of magnetic domains in the sample under 60 Hz, 11.3 AT (peak to peak value) sinusoidal drive field. As seen in these frames, black and white domains and their alteration with the phase of the drive field are clearly observed.

According to the mechanism on the type-II magnetic contrast, these domains are considered to have a magnetization vector parallel to the easy axis [010] which is the horizontal direction in this figure. A large alteration has been observed in these domains with the drive field which has been applied to this direction. On the other hand, another magnetically easy axis is [001] direction (vertical direction) in this sample. Magnetic domains whose magnetization vector are parallel to this [001] direction show, however, no contrast between opposite domains spacing with a 180 degrees domain wall. The determination of the direction of magnetization vector is achieved by 90 degrees rotation of the sample around its normal. Then static observation is achieved to determine the direction of the magnetization vector in each magnetic domain. Magnetic domain walls and the expected direction of their magnetization vectors obtained from the static observation are illustrated in Fig. 4(b). As seen in the stroboscopic images in Fig. 4(a), the progress of magnetization in the present sample almost keeps a flux closure structure with these magnetic domains under the 60 Hz drive field. A comparatively smooth motion is found with 180 degrees domain walls with the alteration of the drive phase. On the other hand, 90 degrees walls where the magnetization alters its direction at right angles are almost fixed on the original position as shown in Fig 4(a).

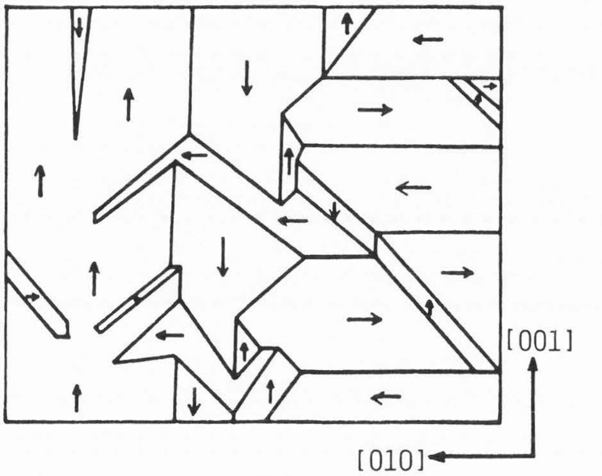
The next example of the stroboscopic observation is the dynamic behavior of the single crystal samples with orientation (110)[001] (GOSS orientation). These Fe-Si platelets are commercially mass-produced by using rolling process as grain-oriented silicon-steel sheets of high permeability. In these steel sheets, single crystalline grains with orientation near (110)[001] are formed along the rolling direction. The degradation of magnetic properties by scratches is one of the important problems to be solved for this material.

In this study, effects of scratching are dynamically observed for a sample with a scratch perpendicular to the rolling direction. The sample used is a ball-point scratched Fe-Si single crystal of 0.3 mm thickness with orientation (110) [001], and the direction of the easy axis [001] is parallel to the rolling direction. In Fig. 5, dynamic observation of this sample under the sinusoidal drive field (60 Hz,11.3 AT) is shown. Fig. 5(a) shows stroboscopic images, corresponding to the phase of the drive field from 0 to 270 degrees in 45 degrees intervals. Three different domain patterns are found in these images. One is a black/white stripe domain pattern which occupies the region apart from the scratch line and seems similar to the stripe domains in the non-scratched samples. Next is a dark strip region which is observed on the scratch line. The width of this strip region is about 3 times wider than the width of the inelastically deformed line as shown in this figure. Finally, some deformed black/white stripe domains are found in the region between the black/white stripe domains and the dark strip region. From these stroboscopic images, it is confirmed that the alteration of the black/white stripe domains under the sinusoidal drive field is caused by the smooth motion of the 180 degrees domain wall. On the other hand, no alteration in the dark strip region is observed from the stroboscopic images with the drive level used in this experiment. As to the deformed black/white stripe region, the alteration of these domains with the sinusoidal drive field is more complicated compared with that of the black/white stripe domains. Deformation, fission and fusion of these domains are observed in these images.

These complicated magnetization processes of each region are considered to be directly related to the distribution of induced stress around the scratch. In Fig. 5(b), magnetizations and the distribution of the stress in the scratched sample are schematically illustrated. The influence of the magnetostriction due to the strong compressive stress around the scratch may cause the magnetization vector parallel to [100] or [010] direction at the region near the scratch line. This region corresponds to the dark strip region in the stroboscopic images. This magnetically altered region may be constructed from fine domains which reduces the magneto-static energy of surface magnetic charges, while these fine domains have not been resolved in this observation. On the other hand, the deformed black/white stripe region is considered to be the region where weak compressive stress remained. The effect of this weak compression results in the production of inner domains of magnetization parallel to [100] or [010] , while these inner domains are covered with the surface domains of a magnetization with [001] direction. The existence of these inner domains results in the complicated magnetization process as mentioned above. It may also contribute to the re-division of the black/white stripe domains, which is important to improve the magnetic losses.

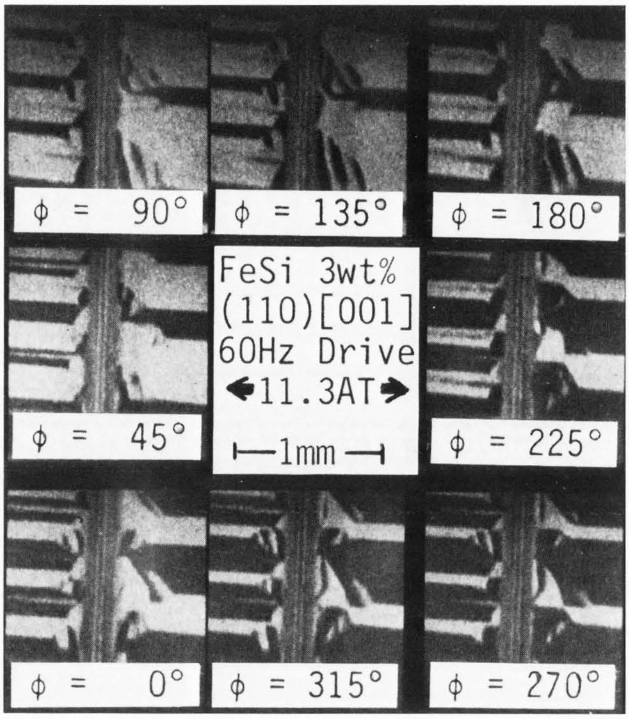
As to the final example of interest, dynamic observation of 'lancet' domains in the sample of GOSS direction is presented. The Fe-Si single crystal with an orientation slightly tilted from (110)[001] shows remarkable isolated lancet-shaped

(a)

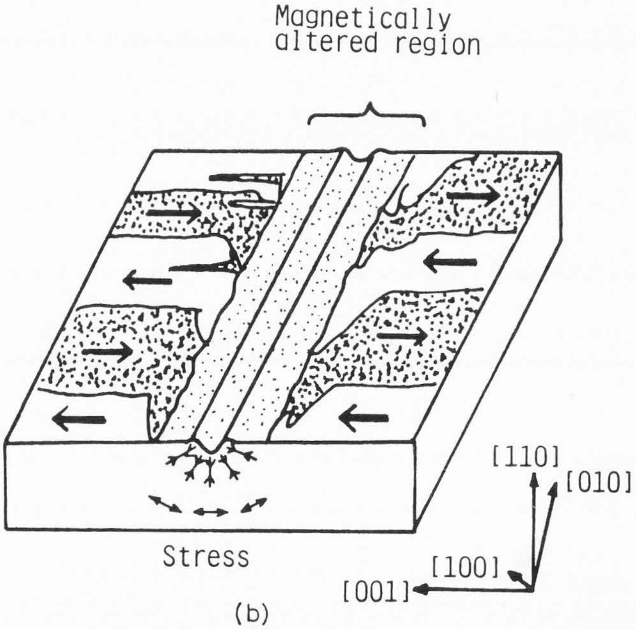


(b)

Fig. 4. Observation of magnetic domains in a Fe-Si 3% single crystal of 0.3 mm thickness with orientation (100)[001]. (a) Dynamic observation under 60 Hz drive field of 11.3 AT at 8-frame mode operation, (b) magnetization vectors in the sample expected from the static image.



(a)



(b)

Fig. 5. Observation of magnetic domains around a ball-point scratch in a Fe-Si 3% single crystal of 0.3 mm thickness with orientation (110)[001]. (a) Dynamic observation under 60 Hz drive field of 11.3 AT, (b) a model of the magnetic structure around the scratch line of [110] direction.

domains in the conventional black/white stripe domains. These domains are usually called 'lancet' domains, and their dynamic response also contributes to the magnetic properties of such materials.

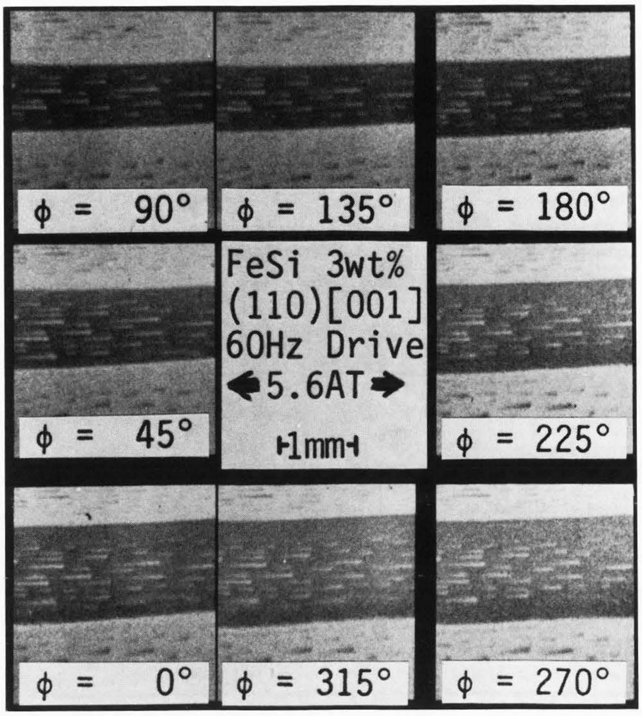
In Fig. 6, the dynamic observation of lancet domains in a Fe-Si single crystal platelet of 0.3 mm with orientation slightly tilted from (110) [001] is described.

In the static observation, it is usually found that these lancet domains are collapsed by the approach of the 180 degrees domain walls and they also reappear after the passage of these 180 degrees walls. These lancet domains are uniformly distributed in the stripe domains for the static field application.

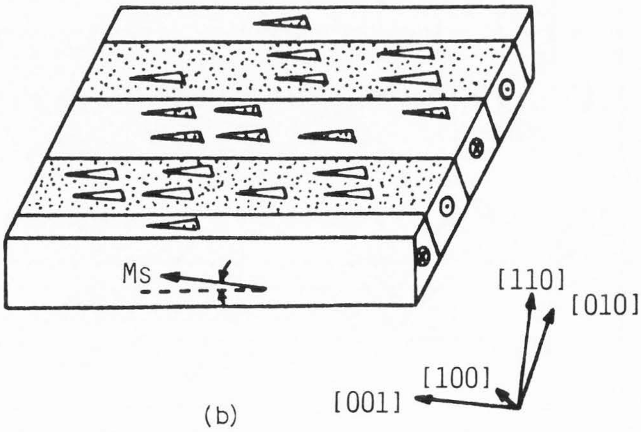
Stroboscopic images in the Fig. 6(a) shows dynamic response of lancet and conventional stripe domains under a 60 Hz drive field of 5.6 AT. The smooth motion of 180 degrees domain walls of the stripe domain is observed in these images. The disappearance of lancet domains within the sweep zone of the 180 degrees domain walls is also found in these images.

From the present observation, it is estimated as a preliminary conclusion that the recovery of the collapsed lancet domains takes more than about 10 ms. For more detailed discussions, stroboscopic observations under the different drive frequency may be desirable.

Fig. 6(b) shows an illustration of the lancet domains in a Fe-Si sample slightly tilted from the GOSS orientation. Although detailed discussions for the origin and the inner structure of this lancet domain have been already reported by several workers (for example Allia et al 1981, Imamura et al 1983), this line-sampling stroboscopic technique can provide very useful information on the dynamic response with the creation and annihilation of magnetic domains in these samples.



(a)



(b)

Fig. 6. Observation of lancet magnetic domains in a Fe-Si 3% single crystal platelet of 0.3 mm thickness with orientation slightly tilted from (110)[001]. (a) Dynamic observation under 60 Hz drive field of 5.6 AT, (b) a domain model of the sample with slightly tilted orientation.

Conclusions

A new stroboscopic technique based on the line-sampling multi-frame stroboscopy in the scanning electron microscopy is developed and applied to the dynamic observation of magnetic domains under the drive field of line frequency used together with a high voltage scanning electron microscope (200 kV SEM, JSEM-200, JEOL Co. Ltd.). Typical examples of the stroboscopic observation of magnetic domains in Fe-Si 3% single crystals with orientation (100)[001] and (110)[001] under the 60 Hz drive fields are presented in this paper.

Present preliminary observations show that this new technique is a very useful tool to analyze the dynamic properties of the magnetic materials under the drive field of line frequency. Therefore this new technique will be widely applied for the improvement of the many practical magnetic materials used in power systems.

Acknowledgments

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The present technique has been developed in cooperation with Professor R. Shimizu and Mr. H. Mase of Osaka University, and the experiment was done in cooperation with Mr. Y. Matsuo, Drs. T. Nozawa and M. Yabumoto of Nippon Steel Corporation. Their support and valuable discussions are gratefully acknowledged.

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Discussion with Reviewers

Reviewer I: Besides magnetic contrast, are there other applications of the line-sampling stroboscopy technique which you can predict?

Author: This new stroboscopy may be applicable to the inspection/testing with the dynamic operation of analog integrated circuits at audio frequency regions by using the secondary electron voltage contrast. The dynamic response of the ferroelectric domains under the alternative electric field/ stress application is also one of the important objects of the present new stroboscopic observation technique.

Reviewer I: What is the limiting resolution of the image under the conditions used for the stroboscopic experiment? It would be useful to state this resolution limit in terms of the width of the smallest domain which could be imaged.

Author: With the slowly altered objects, the spatial resolution of stroboscopic images is just identical with the static case, since the electronic gating of the present stroboscopy has no effect with the probe size or the contrast mechanism. The resolution limit of the type-II magnetic contrast is mainly concerned with the diffusion area of the backscattered electrons. And the diameter of this diffusion area is approximately proportional to two's power of the beam accelerating voltage. At 200 kV operation with Fe-Si samples, a roughly estimated value of the resolution limit is about 10 µm. This value is also altered with the detector take-off angle. In general, lower take-off angle results in higher resolution, while an optimized detector take-off angle is given with the reduction of the topographical contrast.

On the other hand, the motional blurring effectively reduces the spatial resolution with the fast altered objects. Fortunately, the equivalent sampling duration per pixel in the line-sampling stroboscopy can be given to be of very short duration compared with the case of the conventional point-sampling stroboscopy. In practice, the effect of the motional blurring can be neglected in the present line-sampling stroboscopy except for the case of a very fast alteration with the object response.

Reviewer IV: In your explanation of the domain structure in Fig. 5 you mention some supplementary domain structure with magnetization vector parallel to [100] or [010] around the scratch. Could you perhaps visualize this domain arrangement by presenting a schematic illustration?

Author: The domain structure of this region was not resolved in the present observation using 200 kV acceleration voltage. Then it was concluded that the fine complicated domains may be formed in this region to reduce the magnetostatic energy from surface free magnetic charges. An expected magnetic domain pattern of this region is shown in Fig. 7.

Reviewer IV: In the interpretation of dynamic response of lancet domains in Fig. 6 you observe a recovery time of collapsed domains in the order of 10 ms. This observation would mean, that lancet domains are not able to participate in dynamic magnetization processes with frequencies higher than 100 Hz. Have you confirmed this expectation by performing magnetization reversal experiments in this frequency range?

Author: No we have not performed the magnetization reversal experiment at this time, since this observation is preliminary one to confirm the ability of the new stroboscopic technique. The detailed discussion with the participation of the lancet domains to the dynamic magnetization process should be made under the comparison of each experimental result from the dynamic domain observation and the conventional magnetization reversal experiment, since the former observes the surface magnetization process while the latter examines the bulk magnetization process. In the case of lancet domains, there is inner structure of 90 degrees wall except for the surface domain. And it is considered that each contributes to the total magnetization.

Reviewer IV: What kind of surface preparation was necessary for observation of magnetic domains on your sample?

Author: No surface preparation is applied at all with the present samples. The main reason for the requirement of the surface preparation in the SEM observation of the type-II magnetic contrast is to reduce the background contrast from the surface topography. A large magnetic contrast due to the high acceleration voltage of the present SEM makes the surface preparation unnecessary with most of the Fe-Si platelets, even a glass coated (less than several µm thickness) sample. This seems to be the most useful advantage with the high voltage SEM observation.

Reviewer I : D.E. Newbury
Reviewer IV : M. Hastenrath

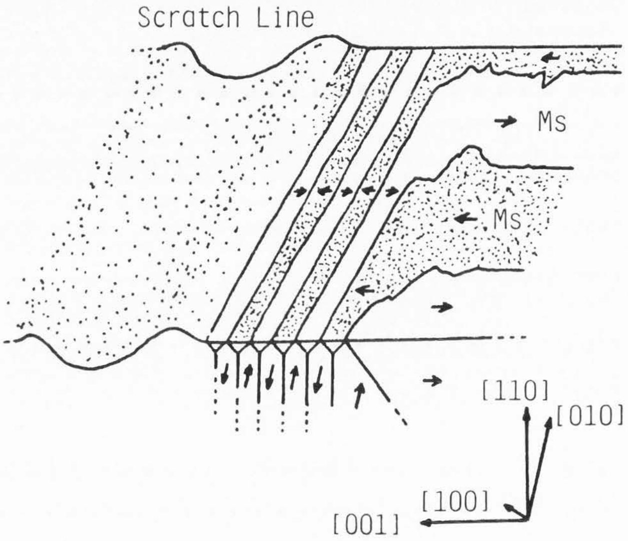

Fig. 7. Illustration of an estimated domain structure around the scratch line of the sample used in Fig. 5.