

HARVARD UNIVERSITY  
DIVISION OF ENGINEERING AND APPLIED SCIENCES

MICHAEL J. AZIZ  
Gordon McKay Professor  
of Materials Science  
Pierce Hall, 29 Oxford Street  
Cambridge, Massachusetts 02138

(617) 495-9884  
Fax (617) 495-9837  
INTERNET: maziz@harvard.edu

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Dr. Reinhardt B. Schuhmann, Associate Editor  
Physical Review Letters  
1 Research Road  
P.O. Box 9000  
Ridge, NY 11961-9000

RE: LE7216  
Dear Dr. Schuhmann:

Enclosed please find a revised manuscript #LE7216, "Nonclassical Smoothing of Nano-Scale Surface Corrugations", by J. Erlebacher et al. We are pleased that the reviews confirmed the importance and broad interest of the work, and that the questions were focused on obtaining more information to assure the validity of the results and interpretation. Clearly we did not have room in the manuscript to present all the relevant information supporting the validity of the work, but we do so in our response below.

The delay associated with our response is due to proper consideration of reviewer A's concern that there may have been sub-surface damage to our samples that was not properly accounted for in the analysis of our results. In order to address these concerns, we have had some of our samples examined in cross-sectional and plan-view TEM, which has taken quite a while. Our conclusion is that although we do observe subsurface damage, it is responsible for at most a minor constituent of the surface relaxation phenomenon we are studying. As such, our results, our conclusions, and the bulk of the manuscript remain unchanged except as to address the minor points raised by the other reviewers comments. These points are addressed in detail below.

*Referee A:*

*This paper describes experimental observations of thermal smoothing of roughened Si surfaces, and their interpretation based on theories discussed in the manuscript. The claim is that roughness decay is non-exponential (contrasting previous experiments by Blakely et al). I have several questions:*

*1) is exponential decay recovered in the temperature range explored by Blakely et al (i.e. above 800 Celsius)? To exclude the possibility of several artifacts slowing down smoothing (see below) it is important to explicitly show that Blakely's results can be reproduced, or -if not- why not.*

The in-situ analytical tool used to monitor ripple relaxation is unable to monitor morphological evolution at temperatures above 800 C because the spectrometer is saturated with black-body radiation. This is explicitly discussed in reference #19 of our revised manuscript, which is a longer paper in preparation.

It might be argued that above 800 C, we could do experiments that involve a fast quench to a low temperature where surface relaxation kinetics are stymied. However, there is a strong scaling phenomenon to relaxation, such that the characteristic time for relaxation goes like the ripple wavelength raised to the 4<sup>th</sup> or 5<sup>th</sup> power. Our experiment takes advantage of the ability to create and anneal structures much smaller than those studied by Blakely, et al.. This means we can probe the interesting physics in the low temperature range but it also means we *cannot* effectively probe the high temperature range. At temperatures of about 1000 C, our structures anneal away in less than 30 seconds, faster than any practical time to do “stop, quench, and look” experiments.

*2) The surfaces studied by Blakely are prepared using non-destructive method. After cleaning in UHV the surfaces are atomically clean, crystalline, and defect free. They are therefore not very reactive (as clean Si surfaces tend to be at elevated temperatures). In contrast, these surfaces are prepared by sputtering, creating extensive subsurface damage not easily removed by annealing (even at 600-700 C). This has possible effects for cumulative surface contamination during the lengthy experiments presented here. Sub surface damage may alter the availability and generation of surface diffusing species, as well as the surface diffusivity itself. Any or all of these effect may have the result of slowing down smoothening relative to exponential smoothening. As argued in the first question, recovering Blakely's results at somewhat higher temperatures than presented here would go a long way to convincing me that these results are in fact meaningful.*

The reviewer raises essentially four concerns here, which we address in turn

a. Possibility of extensive subsurface damage.

The referee suggests that bulk defects may be playing a role in surface relaxation. In order to address this concern, we have performed extensive TEM with the help of Kevin Jones (who is acknowledged), both in cross-section and plan-view. The detailed results of this investigation will be reported in reference #19 of the revised manuscript, and a separate footnote (#17 of the revised manuscript) summarizing the results has been added. We give a more lengthy summary of the results here:

- Newly sputter rippled samples (i.e., unannealed samples in the context of our manuscript) contain a concentration of what are likely stacking faults but might be {311} defects, which penetrate into the bulk a maximum of 10 nm. These stacking faults correspond to a maximum initial concentration of interstitials of  $1e15/cm^2$ . This is the upper bound – if the defects turn out to be {311} defects, then the initial concentration of interstitials will be  $2e14/cm^2$ .

- During relaxation, there is significant reduction of the ripple amplitude of order 5-10 nm. For the results presented in this Letter, amplitude reductions of order 70% are typical. Assuming that all the defects "evaporate" into interstitials which then fill in the troughs (a "worst-case scenario"), we estimate that the interstitials could account for a maximum of only 0.6 nm of the amplitude decrease. Phrased another way, the defect concentration would have to reach  $2.5 \times 10^{16}/\text{cm}^2$  in order to account for the relaxation.

We have added a footnote (#17 in the revised manuscript) to comment on these results. It reads "TEM reveals there are stacking fault defects that penetrate < 10 nm from the surface. However, if all of the observable defects evaporated into interstitials, there would be at most  $10^{15} \text{ cm}^{-2}$  interstitials thereby generated. If all these excess interstitials diffused to the surface and filled in the troughs, it would only account for at most 5% of the typical measured amplitude decrease."

b. Cumulative surface contamination.

We do not believe contamination of the sample played a role in relaxation. There are two potential sources of contamination, (1) hydrocarbon residue from the cleaning process, and (2) metal from the vacuum chamber sputtered onto the sample surface during rippling. Clean, carbon-free samples were a necessary factor in the production of reproducible ripples (and thus good ripples are a measure of sample cleanliness). Samples that were not cleaned properly developed sputter cones that we believe nucleated at silicon carbide contaminated points on the surface. RBS / ion channeling of sample surfaces before and after rippling/annealing does not show any residual metal contamination. RBS does reveal a small amount of Ar, with a concentration of less than 0.1% of a monolayer distributed over a depth of 30 nm. More importantly, within experimental error, this concentration does not change over the course of the relaxation experiment, so this Ar represents an implanted species that does not take part in relaxation.

c. Subsurface damage altering the "availability and generation" of surface diffusing species.

Surface defects from intersection of  $\{311\}$ s or stacking faults with the surface can only enhance the rate of equilibration of the addimer concentration with the bulk. In this case, our analysis, which assumes equilibration, applies even more reliably.

d. Residual ion beam surface damage affecting surface diffusivity itself.

This concern is addressed in detail in response to Referee C's question about anti-phase boundaries.

*3) The data are fitted by a three-parameter fit, raising questions as to the significance of the fit itself. One of the fitting parameters ( $n$ ) could be extracted independently in a rather simple manner by measuring the decay rate vs.  $\lambda$  directly, by monitoring the decay of the light intensity not at a single scattering angle, but at several scattering angles simultaneously. This would demonstrate directly if  $n=4-5$  is reasonable or not. At present, the reader has no choice but to look at the fit, not quite knowing if it makes sense or not.*

While the “fit” contains three parameters, two of them are binary and only one is continuously variable, so the constraints upon each parameter are such that they should not be considered too general. We could just as well call it four one-parameter fits. The binary parameters  $n = \{4,5\}$  and  $m = \{-1,1\}$ . For each of the four combinations of these two parameters, a freely varying activation energy  $\epsilon$  is found.

We compliment the reviewer for his/her insight to measure the decay at several different scattering angles. In fact, we had already done an equivalent measure using LiSSp, in which we measured the spectrum of scattered white light a fixed angle, i.e., the scattering vector is varied in both cases. For the followup long paper (ref. #19 of the revised manuscript), we have examined the data collapse of the spectral decay across this spectrum (see attached figures, #2 and #3, which are taken from ref. #19), and conclude that  $n = 4$  or  $5$  as we have in the paper. (For this particular sample, data collapse with  $n = 4$  looks significantly better than for  $n = 5$ ; however, for other samples the opposite was true). However, this measure is complicated by the fact that we are comparing our results to non-linear models for which Fourier decomposition may not be justified. That is, the OZ model we discuss is applicable only at the “ripple wavelength”, i.e. the brightest Fourier component. It is for this reason that we chose to defer the discussion of this complication, and others like it, to a subsequent paper (reference #19 of the revised manuscript). In the context of the Letter, such a discussion would only distract from the primary results.

*4) How many data sets are actually being presented? Figure 3 gives the impression that there are maybe 3 different measurements, over a rather narrow range of experimental conditions, casting additional doubt on the significance of these results.*

There are 5 data sets represented over a temperature range of about 100 C. We have amended the paper to mention this in the first paragraph on page 7, including a footnote with the wavelengths and relaxation temperatures [reference #22 in the revised manuscript]. For comparison we note that in Blakely’s paper, Ref #14 of the revised manuscript, there are only 10 relaxation time constants presented for a temperature range of 300 C, over an equally limited wavelength range. For our experiment,  $\lambda$  ranged from 350 to 550 nm; for the other experiment,  $\lambda$  ranges from 4 to 6 microns. If we count this way for the papers mentioned by Referee C, Grossman et al. (PRL **74** (1995), 4487), has 4 data sets, Zuo et al. (PRL **70** (1993), 1662) has 7, and Chey et al. (PRL **76** (1996), 3995) has 4 data sets, and the paper mentioned by Referee B (Fu and Williams) has 3. However, for these works, as well as our own, the information presented is really a very small subset of the total information collected in each relaxation run. Our data in fact

contain information about almost the entire spectral decay of the surface morphology, with 2048 channels per spectrum for each point along the decay spectrum. Typically, 30 spectra were acquired during a relaxation experiment, resulting in 300000 data points totaled over all of our samples. We have chosen in this letter to present that subset of our data that represents the most important new physics. More information about the entire set of spectral decay information will be included in our Ref #19.

*The results presented here are potentially interesting, but I don't believe that the authors have presented sufficient, and sufficiently compelling data to make their case. The question raised above need to be answered in detail, with additional data, before this paper is acceptable for publication.*

*Referee B:*

*Development of a non-linear kinetics description of crystalline surface mass transport is the most promising approach to the length-scale bridging problem of stability and evolution of nano-structures. This paper represents an excellent experimental demonstration of the validity of this approach in a particularly problematic case, and thus I strongly recommend that it be published in PRL.*

*Prior to publication however I recommend that the authors address two issues of presentation which I feel are misleading and thus would be a disservice to general readers as well as to those working in the field. These two issues are:*

*1) The issue of attachment/detachment vs. diffusion limited kinetics is a red herring. The authors should not promulgate this artificial distinction by including the conclusion of dimer mediated transport in their abstract. Their conclusion is based on a very weak agreement of their measured activation energy with an estimate of the diffusion activation energy. Since the agreement with the activation energy for attachment/detachment is no worse (their reference 23 and also work by Bartelt and Tromp, PRL 1994 or 5), this is a specious conclusion. Other workers, (Fu et al, Surf. Sci 1997 or 8) have demonstrated that non-classical decay can be fit equally well by ANY combination of a/d and diffusion limited processes, which is probably also the case here.*

At no point did we *conclude* that transport is dimer mediated. It was merely stated to be consistent with our measured activation energy for relaxation. We leave open the option that transport is step-traversal limited, and commented on this toward the end on the paragraph on the middle of page 7. We concede that the original abstract can be misinterpreted to imply that we *conclude* that transport is dimer mediated. We suggest that a wording change may reduce the chance of misinterpretation and change “consistent with an interpretation that dimers mediate transport” in the abstract to “consistent with fundamental energies of creation and migration on Si(001).”

*2) The authors' first sentence of the second paragraph does not adequately (or accurately) represent the significance of their work. The most significant aspect of this work is that they have demonstrated that a decay involving STEP ANNIHILATION can*

*be accurately described by the non-classical step-based model. This is extremely important and should be discussed clearly. On the other hand, this is NOT the first observation of non-classical annealing behavior - Tanaka (Blakely), Bonzel and Fu (Williams) have all observed and properly described non-classical annealing behavior, but NONE of those observations involved configurations with step annihilation.*

We stand by our claim that ours is “the first experimental observation of non-classical annealing behavior for a corrugated surface evolving toward planarity.” We believe that a reasonable interpretation of the meaning of “non-classical” is not simply that morphological evolution is mediated by microscopic step-flow. In order to demonstrate non-classical behavior, we claim that one must show that the macroscopic morphological evolution is inconsistent with the classical models of Mullins and Herring; in this case specifically, one must show that the relationship  $\partial h/\partial t = -B\nabla^4 h$  (which we will call “the M-H equation”) does *not* describe the macroscopic behavior. Tanaka-Blakely and Fu-Williams both measured the decay of metastable structures on Si, examining their results in terms of the microscopic non-continuum step-flow models. However, *neither showed that their results are inconsistent with the M-H equation.* Tanaka et al. discussed the annealing behavior of mounds, not ripples, and did not explicitly discuss the evolution of the height profile. Fu et al. analyzed the relaxation of a periodic 1-dimensionally modulated surface height profile of Si(111) created by step bunching at elevated temperatures. However, their analyses are essentially for the shape of an isolated step bunch. While they do state that the slope does not decay exponentially, we note that the classical model predicts that the width of an isolated feature evolving according to the M-H equation grows not exponentially but as  $t^{1/4}$ , which is entirely consistent with the scaling behavior reported by Fu et al. [see W.W. Mullins, “Solid Surface Morphologies Governed by Capillarity”, chapter 2 of Metal Surfaces: Structure, Energetics and Kinetics (ASM, 1963), pp. 58-59. In fact, Mullins' proof by Fourier decomposition is valid for surface features, such as ridges and grooves, that have zero first moment, but the  $t^{1/4}$  scaling behavior is a quite general result of dimensional analysis which we present in the Appendix below.]

In contrast, our experiment directly measures the Fourier spectrum of the surface morphology as it decays over times much longer than the time for adjacent periods to interact diffusively. We observe that each component does not decay exponentially over long time periods, and thus directly observe inconsistency with the M-H equation.

In order to address the concerns of the referee that our work is placed in the proper context, we propose adding a sentence to the first paragraph reading, “Here, discrete step-step interactions are expected to drive surface relaxation [citing Fu, et al.]”

*Referee C:*

*The authors have studied the decay of a rough Si(001) surface created by ion bombardment. While the experiments are excellent, the authors should provide a more detailed discussion, noting the unique features of the Si(001) surface.*

*The description of annealing of a rough surface has been controversial among theorists and experimentalists for a long time. Although many papers have appeared on*

*the issue, the results have been inconclusive. The analytic theories have been chiefly concerned with the description of the temporal evolution and wavelength scaling properties of the amplitude of a sinusoidal profile. This paper shows measurements of the power spectral density during the decay of a rough Si(001) surface, thereby making a close connection with analytic theory.*

*The authors find an inverse linear decay of the amplitude with time. This observation is used to support the theory of Rettori, Villain (1988) and Ozdemir, Zangwill (1990) (RV--OZ model), which also makes an inverse linear decay prediction. While the observations are interesting, the authors should include in their discussion how well the Si(001) system satisfies the assumptions of the RV--OZ model. This is keeping in view (a) the widely different results observed in the different materials systems (Ge(001), Chey et al., PRL 76, 3995(1996); Cu(001), Zuo and Wendelken, PRL 70, 1662(1993); TiO<sub>2</sub>, Grossman et al., PRL 74, 4487(1995)); and (b) conflicting predictions of different analytic theories (cf. H. Spohn, J. Phys. 13, 69(1993)).*

In (a), the referee mentions a number of papers that have studied thermally-driven coarsening of non-periodic roughness. In each, a lateral length scale (in the plane of the surface) is studied, so the question of non-classical “amplitude” decay is specifically not addressed. In our system, periodicity and the relevant measure being out-of-plane distinguish this study significantly enough from these to not really be comparable. Given space constraints, discussion of these works is just a little too tangential. We will include such a discussion in reference #19 of the revised manuscript.

The work by Spohn and collaborators [HS] (see also Hager and Spohn, Surf. Sci. 324 (1995), pg. 365), on the other hand, is a theoretical attack on the particular amplitude decay problem we are studying. HS present a continuum approach to solving the relaxation of a sinusoidal morphology with a particular eye toward including effects missing from the RV-OZ model to which we heavily compare our results in our manuscript. Specifically, as mentioned in the manuscript, RV-OZ does not incorporate the step-annihilation processes that must occur at the top and bottom of the ripples as they diminish in amplitude. Instead, RV-OZ ignore these “opposite sign” step-step interactions altogether, and surface relaxation proceeds only by the action of step-step repulsion along the slopes of the ripple.

HS used a generalized form of the surface free energy as a function of orientation, based on the Gruber-Mullins model, which they allow to continuously vary along the surface. This is in contrast to the RV-OZ approach in which the chemical potential, whose gradient drives relaxation, is only well defined at discrete step edges. The HS model exhibits a cusp singularity in the surface free energy at the facet orientation. However, HS avoid discontinuities in the solution of the differential equation describing morphological evolution by separating the solution into two regions – the sloping sides of the ripple and the facet at the extrema. By allowing the position of the boundary between these two regions to move as the surface relaxes, they find a mathematically consistent solution to the relaxation problem, both analytically and numerically. In general, HS observe facets to form at ripple extrema, and the slopes of sidewalls become steeper.

There are some problems with HS with regards to our experiment. Primarily, HS predict that the amplitude should eventually decay linearly with time, which we don't

observe. Secondly, by using a continuous form for the free energy, they may be biasing the contribution to overall surface energy reduction during relaxation to the formation of a facet rather than removal of steps. Indeed, the HS model predicts that the step density actually increases along the sidewalls, which we also do not observe. We address these theory concerns by modifying the third-to-last paragraph to read:

“We note that the RV-OZ model neglects step-step interactions across the uppermost and lowermost terraces as well as the overall reduction in surface energy when steps are annihilated. Other models have addressed this issue by analyzing the relaxation along the slopes and along the extremal terraces separately [cite HS]. However, no model except for RV-OZ has predicted a form for the amplitude decay consistent with our data. It may be that a detailed description of the activity at extremal terraces during relaxation is simply not important for this material system under our experimental conditions.”

*The RV--OZ model takes a simple expression for the surface free energy involving an isotropic step free energy  $G_{1}$  and step-step interaction energy  $G_{3}$ , and assumes simple kinetics. How important are features unique to the Si(001) surface, such as*

*(i) The formation of anti-phase domains during sputtering (Bedrossian and Kaxiras, PRL 70, 2589(1993)) . The domain wall motion and the decay of islands nucleated on domain walls can be very slow. Can they slow down decay?*

Bedrossian and Kaxiras (BK) study the rate at which the Si(001) surface damaged by a Xe ion beam heals itself. They observe that small vacancy islands created by the ion beam coalesce into anti-phase domains within a few minutes of annealing, and that a short anneal (6 min) at around 700 C is required to completely heal these surfaces. Our relaxation data were acquired at about these temperatures (650 - 750 °C) but over a significantly longer time (many hours) and any residual surface defect density was produced at significantly higher temperatures than these experiments (> 500 here vs. 350 in BK). Since our 750 C data are at too high a thermal budget to be explained by the BK effect, and since our 650 C decay curves show the same behavior as our 750 C decay curves, we believe that the presence of anti-phase domains does not affect our results.

The question of whether residual surface “damage” in the general form of excess sources and sinks of adatoms can affect relaxation is a generalization of Referee C's concerns expressed by Referee A. Stated in this manner, we see that the question is related to asking whether these defects might not have moved our surface relaxation regime into that of step-traversal (or attachment/detachment) limited kinetics from that of terrace diffusion-limited kinetics where it might be otherwise. Within the context of the non-continuum model in the paper, we have noted that we cannot distinguish between these regimes anyway (since we cannot distinguish between scaling exponents 4 and 5 clearly), and we have made a full discussion of implications for the values the energetic parameters. In any event, the main conclusion of the paper, non-classical relaxation kinetics, would remain unaltered.

Within the context of the classical theory, we might ask whether the presence of excess sources and sinks can affect surface relaxation such that under the classical theory the amplitude decay is no longer exponential in time. We envision two ways in which

the modified surface structure (for the sake of discussion, we will call them "surface defects") can interact with relaxation kinetics in the classical context. First, the defects can speed up equilibration of the addimer gas with the bulk. We have already discussed this case above in the context of surface contamination. Second, in light of the observation of Tanaka et al., ref. #11 of the original manuscript, ref. #12 of the revised manuscript, that step traversal without attachment and detachment is many times as fast as step attachment (about 40 times as fast at 950 C), surface defects could reduce the "mean free path" of the addimers by providing much better sites than steps for attachment, and thereby reduce the surface transport rate. If this were the case, however, then the relaxation rate would *increase* as these kinds of defects anneal away over the course of the experiment. In the classical context, the  $\ln(\text{amplitude})$  vs. time curve would slope downward *and curve downward*, i.e. opposite curvature to what we observe (see Figure 2 of the manuscript).

*(ii) The permeability of steps on Si(001) -- most ad-dimers are believed to hop from terrace to terrace without sticking to steps (Ref. 11). The RV--OZ model assumes incorporation of every atom that reaches a step.*

We note this important aspect of diffusion on Si(001) by carefully referring to step-traversal kinetics, rather than step-attachment/detachment kinetics. Also, our footnote, #21 in the revised manuscript (#19 in the original manuscript), discusses permeation explicitly.

We hope that in light of our response to the reviewers' comments, the manuscript can now be found acceptable for publication in PRL.

Sincerely yours,

Michael J. Aziz

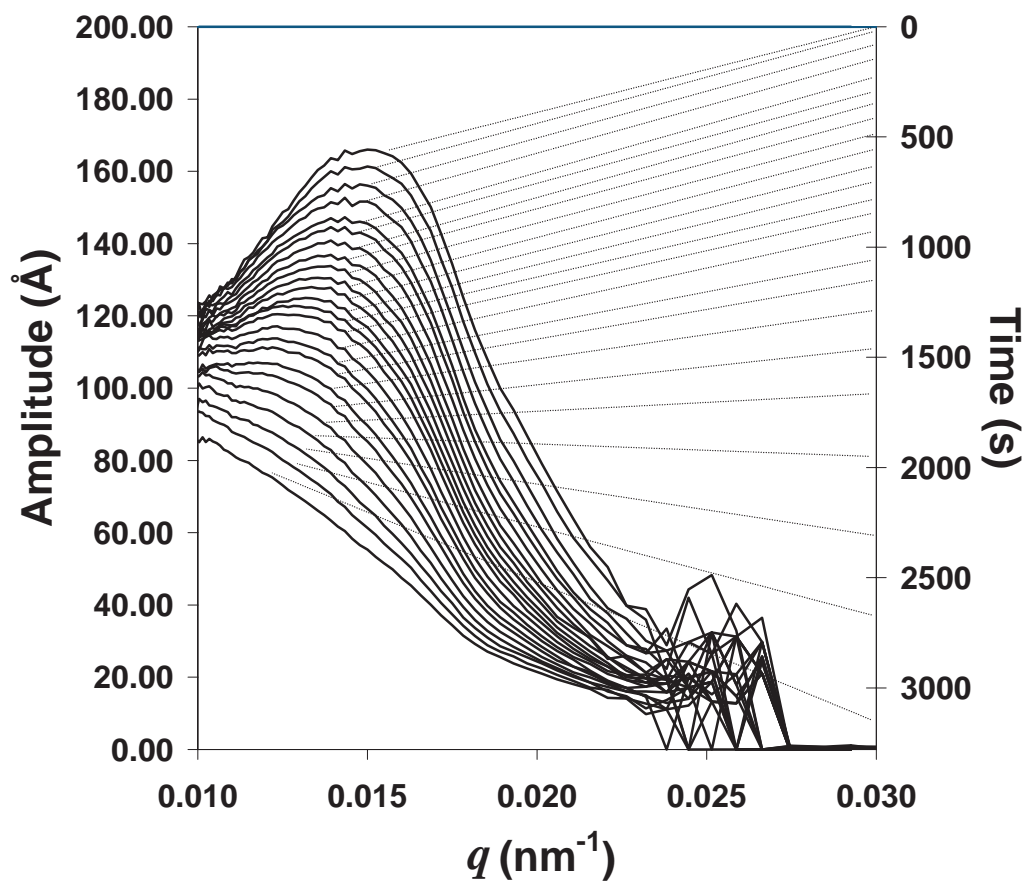


Figure 2. Temporal evolution of the power spectral density for a rippled sample annealed at elevated temperature:  $T = 651 \text{ }^\circ\text{C}$ ,  $\lambda = 405 \text{ nm}$  ( $q = 0.016 \text{ nm}^{-1}$ ).

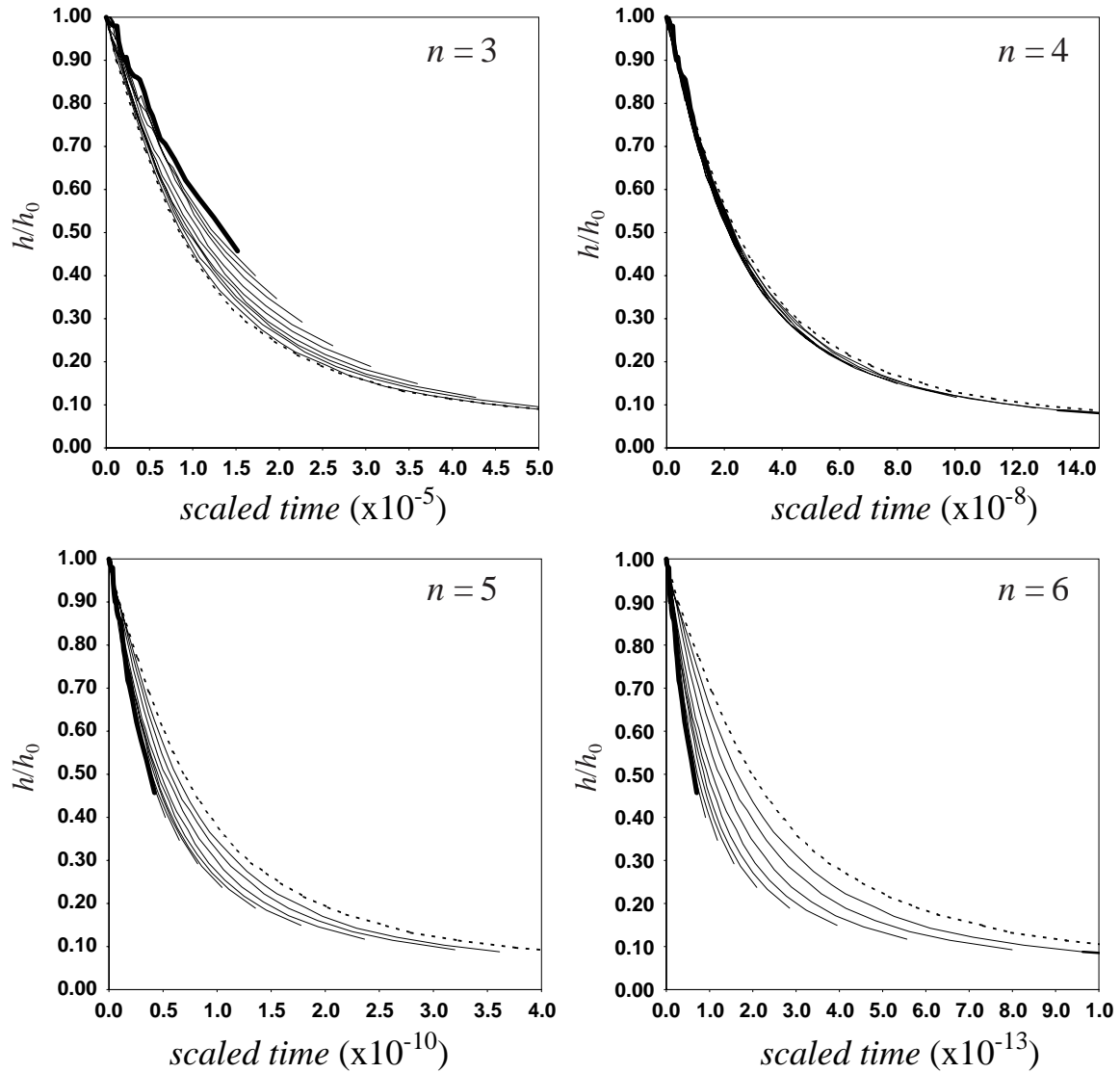
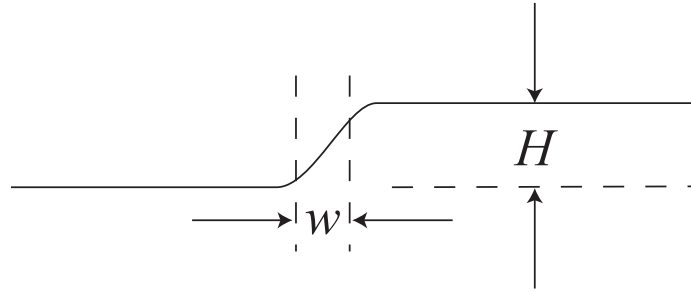


Figure 3. Mode independence of data Figure 2 ( $T = 651$  °C,  $\lambda = 405$  nm). Spatial frequencies of 350 nm (thick dashed line) to 600 nm (thick solid line) with modes every 25 nm are illustrated as representative of the entire relaxation spectrum.

## Appendix

Demonstration by dimensional analysis that the width of a macroscopic step of fixed height evolving according to the M-H equation evolves like  $t^{1/4}$  for long times.

Consider an isolated step, as illustrated. The height of the step is  $H$ , independent of time, and its width is  $w$ . The slope  $\delta = H/w$ , is unitless. The time dependence of the slope will be inverse of that of  $w$ .



Classically, the decay of this step is governed by the equation for the height  $h$  at position  $x$  on the surface. This equation is written in terms of the dimensionless variable  $z = h/H$  as

$$\frac{\partial z}{\partial t}(x,t) = -B \frac{\partial^4 z}{\partial x^4}(x,t). \quad (0.1)$$

Dimensional analysis of this equation shows that the dimensions of  $B$ , denoted  $[B]$  are given by

$$[B] = [L]^4 / [T]. \quad (0.2)$$

Here,  $[L]$  is the length unit, and  $[T]$  is the time unit.

After times long enough that the initial width  $w_0$  has been "forgotten", the width of the step can only depend on the parameters  $B$  and  $t$ . The width cannot depend on  $H$ , as the linearity of the M-H equation has allowed us to remove  $H$  from the problem; note that  $H$  is missing in Eq. (0.1).

For dimensional consistency,  $w$  must be a function of some arithmetic combination of  $B$  and  $t$  whose units are  $[L]$ . The only combination of  $B$  and  $t$  that satisfies this requirement is  $(Bt)^{1/4}$ . That is,

$$w = w(B, t) = f_1(B^{1/4}t^{1/4}). \quad (0.3)$$

One function that satisfies this requirement is

$$w(B, t) = K_1 B^{1/4} t^{1/4}, \quad (0.4)$$

i.e.,  $w \propto t^{1/4}$ , which is our stated objective. To see that this is the *only* possible functional form for the dependence of  $w$  on  $t$ , consider a potential alternative of the form

$$w(B, t) = K_2 B^{1/4} t^{1/4} f_2(B, t, w). \quad (0.5)$$

The function  $f_2$  must be dimensionless. This can only happen if its argument is dimensionless, i.e.,

$$f_2(B, t, w) = f_3\left(\frac{w}{B^{1/4}t^{1/4}}\right). \quad (0.6)$$

We define a new variable

$$u \equiv \frac{w}{B^{1/4} t^{1/4}}. \quad (0.7)$$

Using this, we substitute Eq. (0.7) into Eq. (0.5) to find

$$u - K_2 f_3(u) = 0. \quad (0.8)$$

This equation has some set of discrete roots  $\{u_0, u_1, u_2, \dots\}$ . Evolution of the Mullins-Herring Eq., (Eq. (0.1)) would not jump between roots suddenly, so we conclude that for all time  $u = u_j = \text{constant}$ . From Eq. (0.7) we see that our assertion, that  $w$  behaves as in Eq. (0.4), is proven. Therefore,

$$w \propto t^{1/4}.$$