Cu-Sn Intermetallic Compound Growth in Hot-Air-Leveled Tin at and below 100°C

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ABSTRACT

Low-temperature intermetallic compound (IMC) growth in hot-air-leveled tin (HALT) at lower temperatures was investigated. A technique based on image analysis of backscatter electron scanning electron microscopy images was used to measure the average thickness of Cu-Sn IMC in HALT at 50°C, 75°C, and 100°C with a high degree of precision. The technique made it possible to observe new features of IMC growth in HALT at these temperatures. These measurements demonstrated that the parabolic growth law commonly used to describe IMC growth is not applicable to HALT, particularly at lower temperatures characteristic of storage and service: the conventional parabolic growth rate constant k is not a true constant but is a function of aging time. An alternative two-parameter equation for IMC growth has been proposed: the parameters, true parabolic growth rate K and equivalent preaging time t_o, are true constants that do not vary with aging time. These parameters were determined for two kinds of HALT samples. The activation energy for lattice (bulk) diffusion derived from the true parabolic growth rates K is 11.8 kcal/mol. The growth parameters K and t_o can be used to estimate the solderability shelf life of HALT coatings. The activation energy can be used to calculate the IMC growth acceleration factors for temperatures within the applicable temperature range, and ultimately to develop relevant acceleration aging test treatments.

INTRODUCTION

Use of precoated stock-electroplated tin-lead or hotdipped tin-constitutes a major economic advantage over use of postplating. Hot-air-leveled tin (HALT) variety of hot-dipped tin is a relatively new low-cost connector material that has excellent solderability and formability. Because of its microstructure and composition, HALT is naturally whisker-resistant under most conditions and does not have to be alloyed with lead to prevent whisker growth.¹⁻² The absence of lead in the coating increases the value of scrap and also is a benefit in light of growing environmental concerns. HALT was shown to have a much longer solderability shelf life compared to electroplated coatings.^{1,3,4}

For tin-based coatings the solderability shelf life is ultimately related to the growth of intermetallic compounds (IMCs) at the coating-substrate interface. Preliminary work on Cu-Sn IMC growth rates in HALT demonstrated that these rates are much lower than those in electroplated tin, particularly at temperatures close to the actual storage conditions. The lower rates were attributed mainly to the microstructure of HALT and the resulting prevalence of lattice (bulk) diffusion over the grain-boundary diffusion.

There is little reliable data published on Cu-Sn IMC growth rates below 100°C. This is most likely due to the low growth rates at these temperatures and the difficulty of measuring thin, irregular IMC layers. The commonly used technique that combines optical microscopy with manual measurement of the IMC layer is extremely labor-intensive, depends on the skill and objectivity of the operator, and generally does not offer the precision, accuracy, or resolution necessary for a reliable measurement of a thin IMC layer. Whatever information is available has been measured in electroplated tin or tin-lead; there is no data on HALT. Without knowing the growth rates, one cannot provide an accurate answer to two important questions. First: How does IMC growth affect the solderability shelf life of tin-based coatings, including HALT? Second: What kind of high-temperature accelerated aging procedure is applicable to simulation of IMC growth at lower temperatures?

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This paper briefly describes a technique for measuring thin irregular IMC layers using backscatter electron scanning electron microscopy and image analysis. Using this technique, IMC growth rates were measured at 50°C, 75°C, and 100°C. Because of the high precision of these measurements we were able to observe features of IMC growth heretofore not distinguishable due to lack of resolution. The parabolic growth law used to describe IMC growth is not applicable to HALT, particularly at lower temperatures characteristic of storage and common service environments. This paper proposes an alternative two-parameter equation for IMC growth under such conditions. The parameters are true parabolic growth rate K and equivalent pre-aging time t_o. By applying this description IMC growth acceleration factors can be calculated and relevant acceleration aging tests can be developed.

Samples and Sample Preparation

Two HALT samples were used in the study. Substrates in both samples were phosphor bronze: Alloy 510, .016 inch thick (sample 510), and Alloy 521, .006 inches thick (sample 521). Thickness of tin coatings was measured by X-ray fluorescence, with correction for tin-bearing substrates. Sample 510 is as follows: side A 225 ± 26 microinches; side B: 189 ± 23 microinches. Sample 521 is as follows: side A is 166 ± 22 microinches; side B is 184 ± 26 microinches.

Both samples were aged in Blue M air circulating ovens at 50°C, 75°C, and 100°C. At 50°C and 75°C the aging times were 25, 100, 225, 400, and 625 days. At 100°C, the aging times were 4, 16, 36, 64, 100, and 144 days.

Aged samples were cold-mounted, cross-sectioned, polished, and coated with a thin layer of carbon for SEM work.

IMC Thickness Measurement

The intensity of backscatter electron (BSE) signal of a phase is a strong function of the effective atomic number of the phase. BSE images therefore can be used for measurements of areas occupied by the phases. Figure 1 shows a typical BSE image of a cross-section acquired using image analysis system; for the purpose of easier phase identification the gray scale intensity was inverted. The details of the BSE SEM technique are given in Reference 5. Three distinct regions were observed in all cross-sections: a tin phase, an intermetallic phase (or two phases), and a copper-rich phase (phosphor bronze substrate). The sequence in which the data were acquired was designed to average out any possible variations in magnification during the data acquisition process. Twelve to 24 images were acquired for each sample, temperature, and aging time; this ensured high precision of the measurements. No thickness standards were used concurrent with the measurements; however, SEM was calibrated shortly before the measurements, and both SEM and image analysis system were calibrated after the measurements.

Figure 2 shows a gray-level histogram of the image in Figure 1; there are four peaks in the histogram corresponding



Figure 1. BSE SEM image acquired and stored by image analysis system. Inverse of the original gray scale is used. **a)** tin, **b)** IMC, **c)** Alloy 510. Sample was aged at 100°C for 169 days.

to the four phases in the BSE image of the cross-section. The area under each peak is proportional to the area of a corresponding phase in the BSE image. The two peaks in the middle correspond to the IMC phases; thus the shaded area in Figure 2 is the region of interest (ROI) in the gray scale. After the ROI on the histogram is identified and entered into the image analysis system, the system determines the area of the image that corresponds to the ROI on the gray scale. Figure 3 shows such an area after the BSE image has been processed to remove the noise due to surface imperfections. Next, the image analysis system measured the are fraction $f_{\rm IMC}$ occupied by the IMC. The average thickness of the IMC layer $t_{\rm ave}$ was calculated from the area fraction as

$$t_{ave} = f_{IMC} HW/ML, \qquad (1)$$

where H and W are the height and the width of the image, M is the magnification, and L is the length of the IMC-substrate interface measured on the image. If the interface is parallel to the width, W = L, and

$$t_{ave} = f_{IMC} H/M.$$
(2)

The backscattered electrons originate from a range of depth below the surface; therefore, unlike the optical microscopy where all the information comes from the surface, the BSE signal contains information from a near-surface layer of some finite thickness. This thickness is a function of atomic number, density, and accelerating voltage. In a situation where a thin layer of one phase (phase A) covers the second phase (phase B), the layer of phase A is thin enough for some of the electrons to penetrate it, the BSE signal will be a mixture of electrons from both phases. If most of the backscattered electrons come from the underlying phase B, the area will be considered to be occupied by phase B; this will result in an overestimate of the true area occupied by phase B. In our samples, such a situation could occur at the tin-IMC interface due to the highly irregular nature of the IMC layer. We used a Monte Carlo simulation of electron scattering to determine the variation of the BSE signal (i.e., the backscattered fraction of electrons) with the thickness of the top phase. The computer



Figure 2. Gray-level diagram of the image in Figure 1. Two peaks in the middle correspond to IMC. Shaded area represents the ROI on the scale.



Figure 3. Processed BSE image; this image corresponds to the ROI on the gray scale of Figure 2.

program used was based on a plural scattering Monte Carlo model which is well suited for the case of a thin film over a bulk substrate for accelerating voltages equal or less than 20 KeV.⁶ Two possible cases were considered: (a) tin over Cu₆Sn₅IMC and (b) Cu₆Sn₅IMC over tin.

Figure 4 shows the variation in the backscattered fraction as a function of tin layer thickness over bulk Cu_6Sn_5 . At approximately 0.3 micrometers the backscattered fraction value is midway between the bulk tin value and the bulk Cu_6Sn_5 value. On a gray-scale histogram (Figure 2) this point corresponds to the trough between the tin peak and the IMC peak. Consequently, any area with less than 0.3 micrometers (12 microinches) of tin over IMC will be considered an IMC area. Figure 5 shows the variation of backscattered fraction as a function of Cu_6Sn_5 layer thickness over bulk tin, for this case the midway point is at about 0.25 micrometers (10 microinches). Any area with less than 0.25 micrometers of Cu_6Sn_5 over tin will be considered a tin area. Thus, the minimum observable thickness is relatively small (10 to 12 microinches) and nearly equal for both cases, tin over IMC and IMC over tin. Since a random cross-section will produce an equal incidence of both cases, the error resulting from the first case will be nearly equal and opposite the error due to the second case, and the two will mostly cancel out. For the above reasons, we do not expect a significant error due to the finite depth from which the backscatter electrons originate.

EXPERIMENTAL RESULTS AND DATA ANALYSIS

Figures 6, 7, and 8 show the IMC thickness measurement results for sample 521. Figures 9, 10, and 11 show the re-



Figure 4. Backscattered fraction of the incident electrons at 20 KeV versus the thickness of tin layer over bulk Cu_6Sn_5 . Monte Carlo simulation.



Figure 5. Backscattered fraction of the incident electrons at 20 KeV versus the thickness of Cu_6Sn_5 layer over bulk tin. Monte Carlo simulation.

suits for sample 510. The thickness is plotted versus square root of time; this is the conventional way of plotting IMC growth since diffusion-controlled parabolic growth of IMC is assumed. Also shown on these plots are linear fits to data. Table 1 contains the slopes and intercepts of these fits; the slopes are the conventional parabolic growth rates k. Since linear regression analysis was used to determine the growth rates, a normal distribution test was applied to all data sets; out of a total of 32 sets only 4 deviated slightly from being normally distributed. A t-test was applied to the growth rates and intercepts to determine the confidence limits at 95% confidence level. The confidence limits are listed in Table 1: none of the confidence limits fork include a value of zero. The coefficients of determination for the curve fits (a measure of how good the fit is) are also listed in Table 1.

Cu-Sn IMC Growth in Electroplated Tin and in HALT

Intermetallic compounds grow by diffusion mechanism. The diffusion controlled growth of the intermetallic layer

Table 1. Conventional Cu-Sn IMC parabolic growth rates andintercepts \pm confidence limits at 95% confidence level.Linear fit, IMC thickness vs. t^{0.5}.

Sample	Temperature, °C	Growth Rate, k, µ"/day ^{0.5}	Intercept, W ₀ , μ″	Coefficient of Determi- nation, r ²
521	50	0.56 ± 0.11	20.5 ± 1.7	0.961
521	75	2.6 ± 0.32	17.9 ± 4.8	0.985
521	100	7.8 ± 0.8	15.6 ± 5.4	0.988
510	50	0.52 ± 0.12	21.3 ± 2.6	0.899
510	75	1.9 ± 0.2	21.0 ± 2.6	0.992
510	100	6.5 ± 0.8	16.6 ± 5.7	0.981



Figure 6. IMC growth in HALT over Alloy 521 at 50°C. Averages and standard deviations. Line is a linear fit through the averages.



Figure 7. IMC growth in HALT over Alloy 521 at 75°C. Averages and standard deviations. Line is a linear fit through the averages.



Figure 8. IMC growth in HALT over Alloy 521 at 100°C. Averages and standard deviations, Line is a linear fit through the averages.

of thickness W is described by an equation

$$W = W_0 + kt^{0.5},$$
 (3)

where k is the parabolic growth and W_0 is the intercept. The intercept is zero or small for low temperatures, and it increases with temperature. For electroplated tin over copper it is typically about 1 µm at 100°C and is near zero at room temperature.⁷ Parabolic growth rate k has the same kind of temperature dependence as the interdiffision coefficient, which is

$$\mathbf{k} = \mathbf{k}_0 \exp(-\mathbf{Q}/\mathbf{R}\mathbf{T}),\tag{4}$$



Figure 9. IMC growth in HALT over Alloy 510 at 50°C. Averages and standard deviations. Line is a linear fit through the averages.



Figure 10. IMC growth in HALT over Alloy 510 at 75°C. Averages and standard deviations. Line is a linear fit through the averages.

where Q is the activation energy of the IMC growth and k_0 is a constant. Q and k_0 are parameters characteristic of a specific diffusion mechanism. The activation energy for lattice (bulk) diffusion growth was estimated to be about 15 kcal/mol; the activation energy for grain-boundary diffusion growth was estimated to be only about 6.5 kcal/mol.⁸

Transition from lattice (bulk) diffusion to grain boundary diffusion usually occurs anywhere between 190°C and 80°C, depending on the type of coating.⁸Generally, the smaller the grain size, the higher the temperature of transition.

The above equations give a rather reasonable approximation of IMC growth for common electroplated tin and tin alloy coatings. The growth rate k calculated using equation



Figure 11. IMC growth in HALT over Alloy 510 at 100°C, Averages and standard deviations. Line is a linear fit through the averages.

3 will usually produce an adequate estimate of thickness of an IMC layer at a given time and temperature. However, this equation will produce misleading estimates of growth rate kin case of HALT.

The hot air leveling process takes place at a relatively high temperature. The IMC growth at such a temperature is fast, but the IMC layer thickness is restricted by the competition of growth and dissolution.⁹ Which is why only a thin layer of IMC is present in HALT immediately following the manufacturing process. The thickness of this layer, W_i, usually ranges from 15 microinches to 25 microinches. When HALT is consequently aged at some lower temperature, the presence of an IMC layer at the onset of aging is equivalent to its being aged for some time t₀ before the actual aging starts. This time is equal to the actual time needed for the IMC layer to grow to thickness of W_i in a hypothetical case in which the manufacturing process does not produce any IMC growth. Since due to HALT's large grain size diffusion in HALT is very slow, the linear reaction-limited initial step of IMC growth¹⁰ is likely to be very short, and t_0 would be nearly equal to $(W_i/k)^2$ (from Equation 3). If the aging time is corrected for the time t_0 (which will be called equivalent pre-aging time), then

$$W = K(t + t_0)^{0.5}.$$
 (5)

Figures 12 to 14 show the data for sample 521 with twoparameter curve fits using equation 5; the linear fits are also included for comparison. True growth rate K and equivalent pre-aging time t_0 are the fit parameters. Figures 15 to 17 show the data and curve fits for sample 510. Table 2 contains the fit parameters, confidence limits at 95% confidence level, and coefficients of determination. The coefficients of determination are higher for two-parameter fits (Table 2) than for linear fits (Table 1), indicating a better fit provided by the Equation 5. The confidence limits for the true growth rates K are narrower than the limits for k, also indicating a better fit. The difference between the growth rates k and the true growth rates K is the largest at the lowest temperature, 50°C, and decreases as the temperature increases; i.e., the effect of preannealing is diminished as the aging temperature is increased.

Table 2. True parabolic Cu-Sn IMC growth rates and equivalent times to \pm confidence intervals at 95% confidence level. Two-parameter fit, IMC thickness vs. $(t + t_0)^{0.5}$.

Sample	Temperature, °C	True Growth Rate, K, μ"/day ^{0.5}	Equivalent Time, t ₀ , days	Coefficient of Determina- tion, r ²
521	50	1.12 ± 0.03	388 ± 30	0.998
521	75	3.28 ± 0.12	48 ± 14	0.995
521	100	9.16 ± 0.16	5.7 ± 1.1	0.999
510	50	1.09 ± 0.13	437 ± 153	0.951
510	75	2.66 ± 0.15	90 ± 27	0.990
510	100	7.94 ± 0.12	8.1 ± 1.1	0.999



Figure 12. IMC growth in HALT over Alloy 521 at 50°C. The curve fits are as indicated on the plot.

The important implication of the point that equation 5 is the proper one to use for IMC growth in HALT, rather than Equation 3, is the resulting nonlinearity of the conventional thickness versus square root of time plot: the slope S of the curve is a function of aging time t. Indeed, differentiating Equation 5 with respect to square root of time yields,

$$S = dW/d(\sqrt{t}) = K(t/(t + t_0))^{0.5}.$$
 (6)

The slope S is zero at t = O, and it remains near zero as long as $t \ll t_0$. S is always less than K, and it asymptotically approaches K as aging time t approaches infinity only when $t \gg t_0$, is S nearly equal to K. Therefore, the conventional growth rates k (slopes of the linear tits to data) reported in Table 1 are not true constants; since the slopes of the curves increase with time, so will the slopes of the



Figure 13. IMC growth in HALT over Alloy 521 at 75°C. The curve fits are as indicated on the plot.



Figure 14. IMC growth in HALT over Alloy 521 at 100°C. The curve fits are as indicated on the plot.

linear fits to data. Thus, when the conventional linear fits to the data is used to determine the growth rate, the result will depend on the duration of aging, and the measured rate will be always less than true rate K. This might at least in part account for the scatter in the reported values of the conventional parabolic growth rate k.⁹

The two parameters, K and to fully describe the Cu-Sn IMC growth in HALT on phosphor bronze. Unlike the linear fit approach the two-parameter model allows a reliable extrapolation of IMC growth to higher values of aging time.

The true IMC growth rates determined in this study are summarized in Figure 18. Also plotted are true rates from an earlier work¹ and relevant data from other studies.¹¹⁻¹² The growth rates for electroplated tin are given for comparison with rates for HALT. In the low-temperature



Figure 15. IMC growth in HALT over Alloy 510 at 50°C. The curve fits are as indicated on the plot.





region, at 100°C and below, the true rates for HALT are lower than the rates for electroplated material; the rates are about the same above 155°C for all types of coatings. Figure 18 illustrates behavior common for low-temperature diffusion. In electroplated tin the diffusion mechanism switches at about 155°C. Below this temperature, grainboundary diffusion is dominant in electroplated tin. Above 155°C, the transport mechanism is lattice (bulk) diffusion. For HALT, the diffusion mechanism is the same from 220°C down to 50°C, and possibly down to room temperature (22°C).

The persistence of lattice diffusion in HALT down to low temperatures can be accounted for by the microstructure of HALT, especially by its extremely large grain size.¹ The large grain size means low density of defects, particularly grain-boundaries. The low defect density leads to relative



Figure 17. IMC growth in HALT over Alloy 510 at 100°C. The curve fits are as indicated on the plot.



Figure 18. True parabolic growth rates for Cu-Sn IMC in various tin coatings. Lines are linear fits through data: for grain-boundary diffusion, electroplated tin from 22°C to 125°C; for lattice diffusion, combined HALT (50°C to 220°C) and electroplated tin (155°C to 220°C).

contribution of grain boundary diffusion being insignificant down to very low temperatures. According to the data shown in Figure 18, the activation energy for grain-boundary diffusion is 6.3 kcal/mol; this activation energy is valid for a temperature range of 22°C up to 155°C for electroplated tin. The activation energy for lattice diffusion is 11.8 kcal/mol, and it is valid for a temperature range of 22°C to 220°C for HALT, and 155°C to 220°C for electroplated tin. These activation energies were calculated from the linear fits shown in Figure 18. These values of activation energy are in reasonable agreement with other published values (e.g., Reference 8).

HALT: Solderability Shelf Life and Accelerated Aging

Assuming that no gross deterioration of the HALT coating's surface takes place in storage, the solderability shelf life remains primarily a function of IMC growth. According to the current standard,¹³ as soon as 5 percent of the functional surface area is not wettable by solder, the part fails the solderability test. If the failure was caused by IMC growth, on 5 percent of the surface area IMC is either at the surface or very close to the surface. Knowledge of the relationship describing IMC growth and the growth parameters is therefore necessary to predict the useful solderability shelf life. Equation (5) adequately describes the IMC growth in HALT, so it is possible to predict the thickness of IMC after aging if parameters K and t₀ are known.

The growth rate discussed above is for average IMC thickness; knowledge of it is sufficient for assessment of solderability only if IMC thickness is sufficiently uniform. If the IMC layer is irregular, it is also necessary to know the distribution of IMC thickness. Very little work has been done in this area to date (e.g., see Reference 14). However, using the computer-aided image analysis, obtaining of the thickness distribution is a relatively simple task and can be done concurrently with the measurement of the average IMC thickness. The solderability shelf life then can be estimated as the time at which 5 percent of the IMC is thick enough to reach the surface or to be very close to the surface.

Accelerated aging is supposed to simulate natural aging or certain aspects of natural aging. Since 1964 the accepted procedure for simulating IMC growth in tin and tin-lead coatings is 16 hours aging in dry air at 155°C.¹⁵ This aging was designed to be equivalent to about one year of normal storage. It also has been shown that this test is too severe¹⁶ and that two to four hours of aging compares more favorably with one year at room temperature .^{11,17}

Knowledge of the activation energies for the IMC growth allows for calculation of the acceleration factors for any temperature range within which the activation energies are valid. The acceleration factor for IMC growth for two temperatures T_1 , $T_2(T_1 < T_2)$ within the range can be defined as

$$A(T_1, T_2) = t_1/t_2,$$
 (7)

where t_1 and t_2 are the times for the IMC to grow to a certain average thickness at the corresponding temperature T. Then, according to equations (3) and (4),

$$A(T_1, T_2) = (k(T_2)/k(T_1))^2$$

= exp(2Q(1/T_1 - 1/T_2)/R). (8)

For electroplated tin at temperatures below 155° C, the activation energy Q is 6.3 kcal/mol. With $T_1 = 22^{\circ}$ C = 295K and $T_2 = 155^{\circ}$ C = 428K, the acceleration factor A(22, 155) is about 800. Sixteen hour aging at 155°C will then be equivalent to 530 days at 22°C, or about a year and a half. Therefore, based just on the IMC growth process, the 155°C/16-hr aging is a reasonable accelerated test for electroplated tin to simulate about two years at room temperature. However, based on measurements of deterioration of solderability, other processes such as oxidation make this treatment too severe, which justifies shorter aging times, such as four to eight hours.^{11, 16, 17}

If $T_1 = 22^{\circ}C = 295K$ and $T_2 = 93^{\circ}C = 364K$ such as in steam aging, A(22, 93) is about 60. Therefore, eight hours of steam aging is equivalent to 20 days at room temperature as IMC growth is concerned, which is rather negligible. Thus, in the case of electroplated tin the steam aging test is purely a surface/porosity test and does not involve any significant IMC growth.

For HALT, the activation energy Q for the temperature range from 22°C to 220°C is 11.8 kcal/mol; due to a higher activation energy the acceleration factor for the 155°C test A(22, 155) is much higher, about 270,000. Under these conditions, 16 hours at 155°C is equivalent to about 490 years at 22°C; obviously, this testis much too severe for HALT and its use is absolutely unjustified. For the steam aging test, A(22, 93) is about 2000. Eight hours of steam aging is thus equivalent to about two years at room temperature as far as IMC growth is concerned. The steam aging test therefore appears to be the most suitable and universal accelerated aging test for HALT, testing both surface oxidation and IMC growth. If no steam aging is required but "rather dry heat aging only, the optimal accelerated aging temperature should be chosen and the appropriate acceleration factor calculated, from which the duration of the accelerated aging will follow based on the duration of the natural aging being simulated. Considering that important features of Cu-Sn IMC growth, such as the thickness ratio of Cu₆Sn₅to Cu₃Sn, change with temperature,¹⁸ the accelerated aging temperature selected should be as close as possible to the natural aging temperature, within the practically acceptable limits of the duration of the accelerated aging test.

CONCLUSIONS

A new technique was used to measure Cu-Sn IMC growth rates in HALT at 50°C, 75°C, and 100°C with precision so far not attained in any other comparable measurements. Because of the high precision of these measurements it has become possible to observe features of Cu-Sn IMC growth kinetics previously not distinguishable due to lack of resolution.

The parabolic growth law commonly used to describe IMC layer growth is not applicable to HALT, particularly at the lower temperatures characteristic of storage and service environments. The conventional parabolic growth rate k is not a true constant, but is a function of aging time. An alternative two-parameter equation for IMC growth has

been proposed; it describes the IMC growth better than the conventional parabolic law model does. The two parameters, true parabolic growth rate K and equivalent preaging time t_0 , are true constants that do not vary with aging time in any of the samples.

The Cu-Sn IMC growth parameters were determined for the two kinds of HALT samples and three aging temperatures. These data together with other relevant published data yielded the activation energy for lattice diffusion, 11.8 kcal/mol.

The IMC growth parameter K can be used to estimate the expected solderability shelf life of any HALT coating. The knowledge of activation energies, applied in an appropriate temperature range, makes it possible to calculate the IMC growth acceleration factors for any existing accelerated aging tests and also to develop new acceleration aging tests.

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