Method for measuring the setting process of cement-water mixtures by electrical impedance

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The setting and hardening of cement paste can be taken as the progressive hydration reaction of cement. This paper reports the findings on setting time of cement paste at early hydration by using a novel method named relative electrical impedance (REI). The novel REI method measures the relative electrical impedance between electrodes (ring shaped) embedded in the cement paste. REI measurements were conducted on cement paste samples with a water-cement ratio of 1:2 over 24 hours. REI measurements were compared with VICAT and heat latent methods for indirect validation. It was found that the proposed REI technique determined the 4 periods of the setting time process (sleeping, hydration, deceleration, and diffusion) and allowed the measurement of the sleeping period in detail. It was concluded that the novel REI method could be used as a new method to measure the setting time of cement paste in early hydration.

Introduction

Portland cement is a composite whose chemical composition is controlled by the content of silica (SiO_2), lime (CaO), alumina (Al_2O_3) and iron oxides (Fe_2O_3); these components are finely pulverized and calcined in a rotary kiln where they interact to form a series of more complex compounds (phases) in chemical equilibrium [1]. High temperatures favor the formation of calcium silicate phases such as: C3S tricalcium silicate, C2S dicalcium silicate, C3A tricalcium aluminate, and C4AF tetracalcium alumino-ferrite [1-3]. The compounds C3S (alite) and C2S (belite) are the two hydraulic silicates commonly found in portland cements (75-80% by weight); different contents of C3S and C3A can cause changes in the properties of different portland cements [2].

When only portland cement and water are mixed, it is called a paste, and the water-cement ratio is known as working moisture. The C3S and C3A phases are the most chemically active phases in portland cements, generating a large amount of heat when they react with water, forming known stable hydration products, such as calcium silicate and calcium aluminate hydrates, which produce the hardening of the cement paste. This process is called cement setting [4]. Several stages or periods that take place during the setting process have been identified in the literature and are explained below [3]:

a) **Mixing or dissolution period (I)**: This period corresponds to the cement-water mixture just prepared; the particles of tricalcium silicate C3S and tricalcium aluminate C3A of the cement are in a state of dissolution with the water, thus generating an intense initial chemical reaction generating exothermic reactions which increases the temperature and which ends after 15 to 20 min, due to the formation of a membrane around the C3S and the insoluble hydrated calcium silicate C3A. Duration time: 0 to 20 min.

b) **Sleeping period or nucleation (II)**: This period of inactivity in which water consumption and the amount of hydrates formed is minimal. This relative inactivity is due to the hydrated calcium silicate forming a membrane over the C3S and C3A compounds. With time, the concentration of Ca^{2+} ions in the aqueous phase crosses the membrane of CS3 and C3A until it collapses causing a new hydration. Duration time: 20 min to 5 h.

c) **Hydration or acceleration period (III)**: The formation of the first hydrates begins and, as a consequence, the generation of the porous microstructure increases the mechanical resistance of the mixture; the mixture changes from a gel to a solid. As the hydration of C3S and C3A continues, the concentration of sulfates and aluminates increases, producing a heat peak in the mixture that has its maximum at the end of this stage. The VICAT penetration test and the latent heat test determine this stage. Duration of 5 to 10 h.

d) **Period of gain of mechanical resistance or deceleration (IV)**: In this period, the hydration of the particles continues, developing tubular filaments called fusiform needles of hydrated calcium silicate and hydrated calcium aluminate, which generate greater mechanical resistance. In this period there are also exothermic reactions between CS3 and C3A with the aluminates, generating mono-sulfates. This period begins when the heat of hydration reaches its maximum and ends when the heat of hydration stabilizes at the room temperature, during which time the mixture continues to gain mechanical strength. Duration time:

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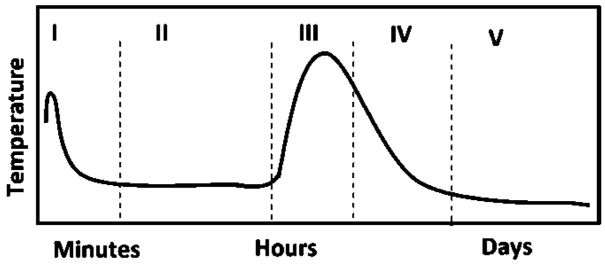

Figure 1. General graph of temperature vs time showing the 5 periods of the cement setting process.

From 10 h to 1 day.

e) **Hardening or diffusion period (V)**: In this period, diffusion rate of ions between hydration products controls the hydration process. The paste hardens and transforms into a resistant solid that cannot be deformed (1 to 28 days). At 28 days, the typical hardening percentage of a concrete is 50 MPa [5]. The strength at 7 days is normally estimated as 75% of the strength at 28 days. Exothermic reactions no longer occur during this period. Time of duration: 1 day to 28 days.

Figure 1 shows schematically the 5 periods of the cement setting process in a general graph of time vs. temperature. It is worth mentioning that in the literature some authors only report 4 periods depending on the characterization technique used [6,7]. Specifically, the hydration (III) and mechanical strength (IV) phases are combined and only one phase of mechanical strength gain is mentioned where the maximum latent heat is present [8]. Other authors do not mention the mixing or dissolution period. [24, 25].

There are several experimental methods to determine the setting of cement, among the most commonly used are: penetration or hardness by the VICAT method (ASTM C191-99,2001) [9,10]; by latent heat (temperature) [4,11]; by electrical impedance [6,12].

The VICAT technique or method measures the penetration of a needle into the water cement mixture. This hardness technique only measures the hydration stage, i.e. the time in which the mixture solidifies. The literature states that for VICAT tests the hardening or onset of setting occurs between 5 and 8 h, depending on the type of cement, the working humidity and the ambient temperature [13]. Although with the VICAT method it is only possible to measure the solidification time of the water-cement mixture, it is a very widely used and accepted test in the literature [14,15].

Another technique for measuring setting is by measuring the latent heat of reaction. In this method, the internal temperature of the mixture is measured and time vs. temperature graphs are obtained where the mixing period, the dormant period, the acceleration period, the deceleration period and the diffusion period can be identified [16-18].

The use of electrical impedance to determine the setting of cement-water mixtures has been reported in the literature. The electrical impedance technique is based on the measurement of the electrical current of the cement-water mixture when a voltage is applied at a given frequency. It is reported that it is possible to measure by electrical impedance the dormant period, the hydration or acceleration period, the mechanical strength or deceleration period and the hardening or diffusion period [16, 19]. These authors report impedance measurements during setting using a capacitor-type structure, i.e. metal-mixed-metal, and monitor the impedance for 28 days [6]. From the Cole-Cole plots obtained, they calculate the equivalent circuits and model the setting kinetics [16, 11, 12, 20]. Although electrical impedance is a method to measure the setting process, there are few reports in the literature that have measured early stages of the setting process [21, 22]. The electrical impedance technique uses the capacitor-type structure to measure. The capacitor structure is when the electrodes are used as walls and in between them is the water-cement mixture. This capacitor structure causes several problems in the impedance measurement such as: a) Shielding of the impedance value when the impedance value is very low (20 to 100 Ω); b) Partial loss of electrical contact between the mixture and the electrodes when the acceleration process starts due to the solidification of the cement-water mixture. Some authors report electrical impedance measurements with a high standard deviation in samples with 24 days of setting [6] due to the loss of electrical contact by breakage of the cement matrix, and therefore, by loss of contact of the electrodes.

To avoid the problems associated with the capacitor structure, in this work it is proposed to use 3 small ring-shaped electrodes placed at different depths within the water-cement mixture. Since the electrodes are at different depths but equidistant, the impedance value measured between electrodes is relative and will be correlated to the chemical and hydration changes of the water-cement mixture in the regions where the electrodes are located. We call this new technique Relative Electrical Impedance IER. It is worth mentioning that this way of measuring electrical impedance has not been used in the literature on cement, so this is the first time it has been reported.

Therefore, the present research aims to use the new technique called Relative Electrical Impedance IER to measure the setting process of cement-water mixture during the first 24 hours. The IER results will be compared with the accepted VICAT and latent heat (temperature) methods to discuss the setting process.

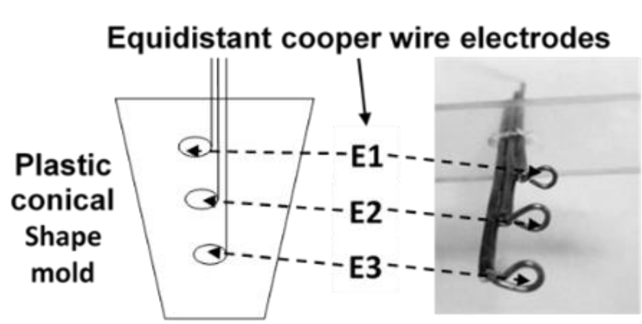

Figure 2. Probe with electrodes a) schematic diagram b) arrangement of electrodes constructed.

Materials and Methods

Cement-water mixtures

Composite Portland Cement (CPC30R) manufactured by CEMEX (Mexico) and distilled water were used to make the mixtures. The cement-water ratio used to make the paste was 2:1, i.e., for every 100 grams of cement, 50 milliliters of distilled water were used. The mixture was placed in a conical plastic container 10 cm high and 8 cm in diameter with a capacity of 350 ml (see Figure 2). The measurement conditions were at room temperature.

Relative Electrical Impedance measurement

The test tube consisted of a conical-shaped plastic container of approximately 350 ml with a height of 10 cm, a lower diameter of 5 cm and an upper diameter of 8 cm. The 2:1 cement-water mixtures were introduced into the container and the electrodes were placed. Acrylic bases were built to hold the electrodes as shown in Figure 2. The electrodes used are made of commercial insulated copper wire number 16. The length of the electrodes is 20 cm and the end that is introduced into the mixture is removed from the plastic insulator and given a circular ring shape (see Figure 2). The circular ring shape is to guarantee a measurement area and minimum contact loss during the drying process of the mixture. The electrodes inside the specimen are vertically and at an equidistant distance of 1 cm as shown in Figure 2.

Measurement of the setting process

The hardening process of the water-cement mixture was measured by means of: a) Relative Electrical Impedance Method measuring the impedance between equidistant internal electrodes b) Latent heat method measuring the internal temperature of the mixture and c) VICAT method (penetration as a function of time). The comparison between these three methods with their respective curves as a function of time will allow correlating the physical changes that occur in the hydration and hardening process of the water cement paste with the associated chemical reactions reported in the literature. It will also be possible to validate the proposed IER technique by comparing the information between each technique in relation to the setting process.

Hardness Measurement (VICAT Method)

The VICAT method basically consists of performing penetrations at different times with a metal needle supported on an overweight. A commercial VICAT attachment that complies with ASTM C191 [9] was used for the measurement. A 6.5 cm long needle with a diameter of 1 mm was used and a counterweight of 300 g was used. A position sensor (resistive potentiometer) was incorporated into the VICAT attachment in order to capture, by means of the PC, the penetration distance of the needle. The penetration measurement was taken every 10 min. The measurement units used were normalized. Figure 3 shows the measurement arrangement used.

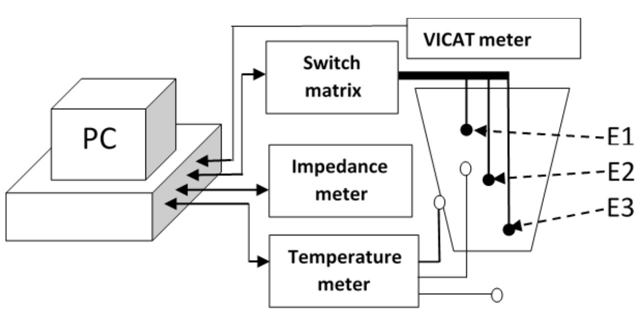

Figure 3. Schematic diagram of the array used to measure Relative Electrical Impedance.

Latent Heat Method (Temperature Measurement)

The experimental setup used to measure the latent heat during the cement setting process is shown in Figure 3. Three temperatures were measured: the internal temperature of the sample, the temperature at the mold surface and the ambient temperature. T-type thermocouples were used to measure the 3 temperatures. Figure 3 shows the position of the 3 thermocouples in the experimental setup used. A National Instruments model 9211 4-channel differential temperature meter was used.

Relative Electrical Impedance Method

The proposed relative electrical impedance method called IER is developed to measure the early stages of setting (0 to 22 h) where the specimen has sufficient moisture to measure electrical impedance without loss of electrical contact. The proposed method is basically based on the measurement of the electrical impedance modulus Z at 3 different depths of the specimen. For this purpose, 3 electrodes (E1, E2 and E3) are introduced on the vertical of the specimen and at an equidistant depth (1 cm between each electrode) (Figure 2). The electrical impedance between electrode E1 and electrode E2 is measured and we call it Z1-2, then the impedance between electrode E2 and electrode E3 is measured and we call it Z2-3 and finally the impedance between electrode 1 and electrode 3 is measured and we call it Z1-3. To measure the electrical impedance, a HIOKY LCR Hi-Tester Model 3532-50 impedance bridge was used.

The value of Z is calculated by means of the equation

$$Z = \frac{E_t}{I_t} = \frac{E_0 \, exp\,(jwt)}{I_0 \, exp(jwt + \emptyset)} = \frac{E_0}{I_0} \, Cos\, \emptyset + j \, \frac{E_0}{I_0} \, Sin\, \emptyset = |Z| Cos\, \emptyset + j\, |Z|\, Sin\, \emptyset$$

The value reported in this work is the modulus of the impedance |Z| at a working voltage of 1 volt, a limited current of 1 mA and a frequency of 10 Khz. In preliminary impedance measurements, it was observed that frequency values higher than 100 Khz, also increases the conductivity and the noise due to the less input impedance of the device used for measurement. The value of 10 Khz was determined by means of preliminary impedance measurements, seeking that the impedance value will not present variations greater than 1%, in addition to the fact that at high frequencies (greater than 1Khz) the double layer effect is avoided. In the International System of Units, electrical impedance is expressed in Ohms (Ω). In the literature it is found that the Z-impedance value for water cement mixtures is of a low value (20 to 100 Ω) [6, 16] due to the fact that cement has many ions and minerals, as well as iron oxides. The impedance value depends on the amount of water contained (water:cement ratio) and the type of cement used.

A device called "switch matrix" was developed to connect the HIOKY impedance meter with each electrode independently, so that it is possible to measure the electrical impedance Z1-2, Z2-3 and Z1-3 independently.

A special computer program was developed to plot at the same time the electrical impedance between electrodes, the internal temperature of the mixture, the temperature on the wall of the specimen, the ambient temperature and the VICAT penetration value. Figure 3 shows the experimental setup used for temperature data acquisition, IER measurement and VICAT penetration.

Results

The relative electrical impedance of water-cement mixtures was measured at a 2:1 wetness ratio. Figure 4 shows the graph obtained of the relative electrical impedance Z1-2 and Z2-3; the graph of the internal temperature of the sample and the graph of the Vicat penetration test. The measurement time was 24 h.

Penetration Test (VICAT)

Figure 4 shows the measurement of penetration (hardness) by the VICAT method where 3 stages are defined: A first stage from 0 to 5 h where the needle penetrates completely into the mixture; A second stage between 5 to 10 h where the water cement mixture begins to harden so the penetration decreases until it does not penetrate indicating that the water cement mixture has solidified. From this moment on (10 h) there is no penetration because the mixture has hardened. This would be the third stage (10 h onwards). In the literature, the hardening of the water-cement mixture is associated with the period of gain of mechanical resistance or acceleration and is due to the hydration of the tricalcium silicate.

Latent Heat (Temperature)

Figure 4 shows the measurement of the internal temperature of the sample. The graph defines 3 important stages: A first stage from 0 to 2.5 h, where an initial temperature of 28 ºC and a final temperature of 26 ºC is obtained. The initial temperature of 28 ºC is due to the exothermic reaction when mixing cement with water. A second stage of 2.5 h to 10 h where the temperature increases to a maximum of 34 degrees due to an exothermic hydration reaction. A third stage (10 to 22 h) corresponding to the temperature decrease until reaching room temperature (approximately 24 h). In the literature, the internal temperature of the water-cement mixture is associated with the latent heat of the chemical reactions of the hydration process. Note that there is a relationship between the measurements of the 3 techniques. It can be clearly observed that the highest temperature reached (highest peak temperature) is related when the needle no longer penetrates (mechanical strength) corresponding to 10 h of setting.

Relative Electrical Impedance

The initial values of relative electrical impedance were 41 Ω between electrodes 1-2 (Z1-2), 42 Ω between electrodes 2-3 (Z2-3), and 46 Ω between electrodes 1-3 (Z1-3). It is observed that the measurement of the relative electrical impedance has 3 stages with significant slope changes: A first stage that goes from 0 min to 5 h with low values of electrical impedance (30 to 50 Ω); a second stage that goes from 5 to 10 h and corresponds to the increase of the electrical impedance from 50 Ω to approximate values of 400 Ω; and a third stage that goes from 10 to 22 h where the approximate value of impedance is 2800 Ω. In the literature it is reported that the impedance measurement can reach values of MΩ in about 28 days [6,12].

Figure 5 shows the relative electrical impedance Z1-2 and Z2-3 from 0 to 5 h approximately and the following behavior is observed: There is an initial value of electrical impedance of 52 Ω, which decreases rapidly to 45 Ω during the first 20 min; after 20 min to 2.5 h the impedance continues decreasing but more slowly until reaching a minimum of 40 Ω which then increases slowly to a value of 45 Ω at 5 h. Subsequently, an abrupt increase in impedance is observed which corresponds to the acceleration period and ends at 10 h according to Figure 4. The abrupt increase in impedance during the acceleration period corresponds to the hydration

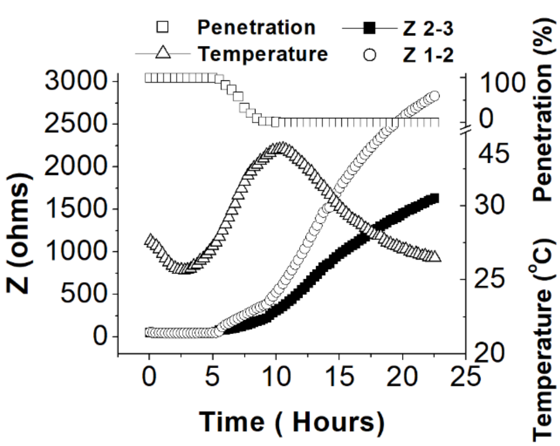

Figure 4. Plot of impedance Z1-2, Z2-3, temperature and penetration (VICAT) versus time of a 2:1 moisture ratio water cement mixture.

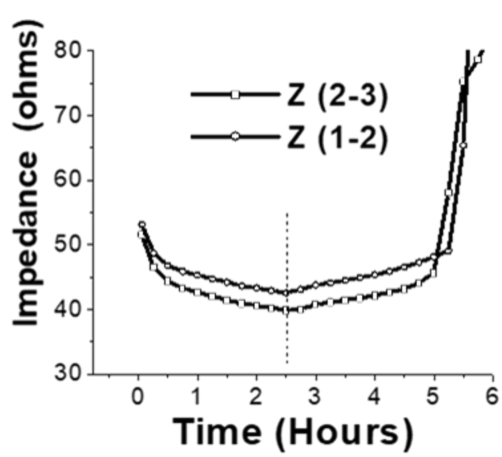

Figure 5. Relative electrical impedance Z1-2 and Z2-3 in the period from 0 to 6 h.

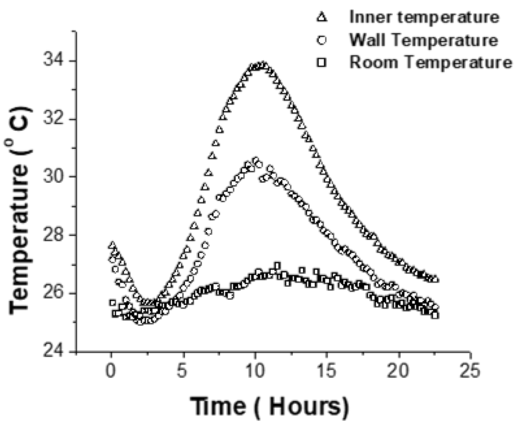

Figure 6. Internal, wall and ambient temperature of water-cement mixture.

of C3S and C3A so it is the beginning of the hardening of the mixture or gain of mechanical strength [23]. There is a strong correlation between the IER and the VICAT penetration measurement.

As the setting process is dependent on the ambient temperature, Figure 6 shows the internal temperature of the specimen (temperature plotted in Figure 4), the temperature of the specimen wall and the ambient temperature. It is observed that the ambient temperature only varies 2 degrees in the 22 h reported, while the internal temperature reached a maximum of 34 ºC and the temperature of the specimen wall reached a maximum of 31 ºC. This indicates that the internal temperature measured corresponds to the latent heat of the reaction and not to changes in the ambient temperature.

Discussion

According to the literature, during a setting process, there are different periods and chemical reactions forming different compounds. According to the results shown in Figure 4 and Figure 5 we can say that the new method reported in this work corresponding to the relative electrical impedance IER is able to measure the following periods in a setting process of cement-water mixture:

a) From 0 to 10 min is when the cement and water are mixed; the impedance gives an initial high value of 52 to 54 Ω. This corresponds to the mixing period.
b) From 10 min to 2.5 h, the relative electrical impedance decreased to 40 Ω (Figure 5) indicating a dormant period where the water-cement mixture becomes a gel inhibiting hydration. According to the latent heat measurement, this period corresponds to the lowest temperature value. This period corresponds to part of the dormant period.
c) From approximately 2.5 to 5 h, the IER increases to 45 Ω (Figure 5) indicating the onset of Ca^+ diffusion and therefore the increase in impedance. The impedance value increases but remains at low values indicating that the aqueous phase still exists (because of the low impedance values). For the latent heat measurement this period corresponds to a slight temperature increase. This period corresponds to part of the dormant period.
d) From 5 h to 10 h the mechanical gain period occurs where the mixture obtains the necessary hardness so that the VICAT needle no longer penetrates. In this period the maximum latent heat is reached (10 h). The impedance value undergoes an abrupt change (from 50 to 500 Ω) corresponding to the hydration period where the mixture becomes a solid paste. There is a decrease in humidity which causes the impedance to increase. It is mentioned in the literature that the gain in mechanical resistance is due to the formation of hydrated tricalcium silicate; hydration generates exothermic reactions increasing the temperature and decreasing the humidity due to hydration. This period then corresponds to the Acceleration Period.
e) The hardening period of the paste occurs from 10 to 22 h. The VICAT test no longer measures (does not penetrate the needle) because the hardness of the mixture is very high. The temperature begins to decrease in value because the chemical reactions associated with hydration no longer generate latent heat. The impedance value increases with increasing slope (500 to 2500 Ω) indicating less moisture due to the formation of tricalcium silicate hydrate and tricalcium aluminate hydrate compounds. This period then corresponds to the de-acceleration period.
f) From 22 h to 28 days: The impedance value increases slowly according to the controlled evaporation of water by diffusion, which generates a resistant and hard solid. During this period, the water-cement mixture hardens, which corresponds to the diffusion or hardening period.

From the above results it can be concluded that the proposed IER technique can measure the 5 periods that occur in the setting of cement according to the literature: mixing, sleeping, acceleration, de-acceleration and diffusion. However, according to Figure 5 it is observed that the impedance decreases to a minimum at 2.5 h and then increases up to 5 h. This behavior is also observed in the latent heat (Figure 6). There is no discussion in the literature about this behavior. We believe that this behavior can be associated to the mechanisms of the hydrated surface layer (decrease of the impedance value due to the decrease of ions) and to the increase of Ca^+ ions that perforate the hydrated layer (increase of the impedance value due to the increase of ions). At the moment that the calcium ions perforate the hydrated layer, the acceleration period begins, where the main mechanism is hydration, so that the impedance values increase considerably, indicating a different behavior from the dormant period (5 h).

As an important result we can say that this is the first time that the dormant period (using the IER technique) can be experimentally measured in detail. The observed behavior and the corresponding discussion will be the subject of further research in the future.

Conclusions

A new technique called Relative Electrical Impedance IER was applied and compared with the latent heat technique (internal temperature of the mixture) and with the VICAT technique (penetration) as a function of time for water cement mixtures. It is concluded that the IER technique can be used as an alternative technique to measure the early setting of water-cement mixtures.

The proposed IER technique is a technique that allows measuring the different periods or stages that occur in the setting process of cement-water mixtures in an accurate and reliable way. The IER technique allows measuring the 5 corresponding setting periods of cement-water mixtures: Mixing, sleeping, acceleration, deceleration, and diffusion.

The IER technique identified two behaviors in the dormant period; and consistently, the latent heat measurement also identified this behavior. Further experimentation is needed to associate this behavior with possible nucleation.

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