

FeO₂3

The Many Faces of Iron: An Exploration in Cooling

by Dr. Carol Marians

One of the more fascinating, sometimes frustrating parts of ceramics is learning to balance the innumerable factors that affect the outcome of a firing. Glaze ingredients, the clay body used, firing cycles, atmospheres, kiln-stacking techniques and geography (to name a few variables) can all affect firing results.

This may be frustrating if you don't control those variables, but if you do, there is opportunity for new discoveries. By changing just one variable, the same glaze recipe can be deliberately manipulated to yield different results. In this instance, I decided to investigate one variable in an iron-rich glaze: the cooling period.

I achieved greatly differing results in a single glaze with a single clay body, consistent glaze thickness and application, and the same heating schedule for all of the firings. The differences in the resulting appearance of the glaze on the pots came exclusively from their heat treatment *after* they reached maturity.

When the witness cone bends, the glaze should be fully vitrified. The kiln has reached temperature, but has not yet begun to cool. I studied what happens between that point and the return of the kiln to room temperature. I found that I could get a glossy black surface, a densely textured rough surface, a golden red/mud color, or anything in between, just from different cooling schedules.

How does this happen?

At the top of the firing cycle, the glaze is matured, but not watery; it doesn't flow

off the pot. At this point, the glaze is not a homogenous melt, but a mixture of several melts. It is not fully blended. It may contain a dissolved second phase—in our case an iron compound—analogue to sugar dissolved in hot tea. More sugar dissolves in hot tea; less as the tea cools. The sugar precipitates as crystals as the tea cools. Our glaze, when melted, has a dissolved iron compound—the “sugar” in the tea. The iron precipitates as the glaze cools. So how does the iron form in the glaze?

Glaze is more complex and more viscous than tea, inhibiting motion. The iron crystals cannot precipitate and sink to the bottom of the glaze, nor can they grow very large, as the iron ions do not congregate in the same location. Instead, as the glaze cools, the dissolved iron separates out, forming numerous small crystals suspended in the glaze. The number of particles, and their eventual size, is affected by the surface texture of the underlying clay body, the cooling speed of the melt, the thickness of the glaze application and several other factors. The competition between the number and size of particles as the glaze cools results in the variety of desirable effects (see accompanying figures).

As it cools, the glaze becomes progressively more viscous and less mobile, until it reaches a temperature at which it “freezes” and nothing can move or precipitate within it. If the glaze is held at a temperature high enough to permit continued mobility of the iron into progressively larger crystals, but low enough

recipe

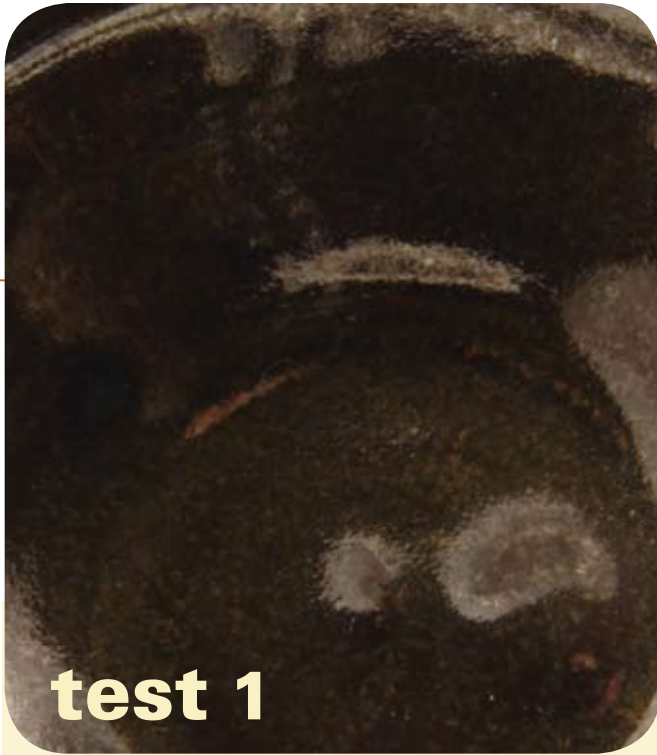
The glaze used in these tests is a minor modification of the glaze GA16 from Michael Bailey's *Cone 6 Glazes*, poured thick on Georgies Ceramic Supply's G Mix 6 clay body.

GA16 VARIATION (Cone 6)

Bone Ash	4.6 %
Dolomite	13.6
Lithium Carbonate	4.6
Red Iron Oxide	9.1
Unispar	22.7
Bentonite	1.8
OM4 Ball Clay	20.9
Silica (Flint)	22.7
	100.0 %

EMPIRICAL FORMULA

CaO	0.4126
K ₂ O	0.0454
Li ₂ O	0.2013
MgO	0.2521
Na ₂ O	0.0886
Al ₂ O ₃	0.3424
SiO ₂	2.7566
P ₂ O ₅	0.0480
Fe ₂ O ₃	0.1912
TiO ₂	0.0104



test 1

Cool down: A continuous cool from Cone 6 to 1500°F at -150° per hour.

Results: This is the cool-down profile from Hesselberth and Roy. It gave a predominantly glossy black glaze, not greatly different from the quick cool, but with a hint of variegated color. I could see isolated metallic bronze and red flecks, but no crystals breaking the surface.

Cool down: An uncontrolled drop from 2200°F to 1750°F, then -50° per hour from 1750°F to 1500°F.

Results: The cooling was slower from 2200°F down to 1450°F. Because the solubility of iron in glaze decreases at lower temperatures, I cooled at 1/3 the speed between 1750°F and 1500°F. The result was a substantially textured surface, with much visible variation, and crystals of a variety of colors breaking the surface. The glossy black was gone, and the surface variation uniformly distributed. There were a relatively small number of largish particles. The color was intermixed red, bronze and mud brown. Bronze predominated where the glaze was thickest. I interpreted this as substantial particle growth below 1750°F, with little precipitation of new particles.



test 2

that the glaze doesn't run off the pot, the surface will become matt. The multitude of tiny iron particles disrupt light transmission. Otherwise, the glaze solidifies with the same smooth, glossy surface as it had while fully melted. If the glaze is cooled quickly, few visible, very small particles form. Most of the visible color is the reflection off the smooth surface. This gives an aesthetically pleasing clear glossy black glaze, somewhat akin to a temmoku (see test 1). The opacity and depth of the glossy black show that the glaze can dissolve quite a lot of iron.

As the glaze cools and becomes more viscous, crystals begin to form at edges and imperfections in the body. If the glaze layer

is thin, different kinds and shapes of crystal will form. If the crystals are stuck to the clay body at the bottom of a thick opaque glaze layer, they will be largely invisible. Crystals that float on top of the glaze give the appearance of sandpaper, which can present utilitarian problems. We want the crystals near the surface but not on it, large enough to create surface and color effects, but not be overwhelming.

A series of cool-down profiles with lots of jigs and jags showcases a different phase, exposing a range of surface effects. This translates into profiles with one or more narrow temperature ranges with extreme slow cooling and/or long holds, and possibly no retarded

cooling outside the selected ranges. Since extended firing cycles can be costly, I framed my experiments with a maximum extension to the firing cycle of four hours.

I started out with the firing profile in Hesselberth and Roy's *Mastering Cone 6 Glazes*. The ramp for reaching temperature was a fast rise (200°F in the first hour, then 500°F per hour to 2100°F) until the last three hours, which had a rise of approximately 30°F per hour. Orton cones showed a hard Cone 6. These firings were done in a very old Skutt 1227 with a computer controller. I examined the results of my firings and based my next firings on those results, only changing one factor with each firing. I chose 1450°F as



test 3

Cool down: An uncontrolled drop to 1750°F, then -50° per hour to 1600°F, a hold at 1600°F for one hour, then -50° per hour to 1500°F.

Results: By adding a one-hour hold at 1600°F, the color shifted from gold/brown to red/gold. The red and brown regions followed the throwing lines, indicating that glaze thickness has significant influence. The strength of this effect showed there is a critical region for this glaze's development somewhere near the temperature 1600°F.



test 4

Cool down: An uncontrolled drop to 1750°F, hold at 1750°F for half an hour, then -50° per hour to 1650°F, hold at 1650°F for one hour, then -50° per hour to 1500°F.

Results: Adding a half-hour hold at 1750°F and a one-hour hold at 1650°F gave smaller particles and a near-smooth, lustrous satin, variegated bronze glaze with small specks of red and brown. The original glossy black was completely gone. Color variation in the throwing line showed the considerable effect that glaze thickness has. The half-hour hold at 1750°F facilitated the formation of a large number of small particles, leaving little free iron to add to crystal growth later. This uniform result was much like a pointillist painting, with exceedingly fine points. Moving the hold from 1600°F up to 1650°F could have a similar effect. Alternatively, we could see this change as a result of the glaze spending more time in the critical temperature interval for crystal development.



test 5

Cool down: An uncontrolled drop to 1800°F, then -50° per hour to 1450°F.

Results: As the previous test result could have come from extended time in the crystal growing range, or specifically from the hold at 1650°F and 1750°F, I gave this firing just as much time in the sensitive zone, but uniform decrease in temperature over the extended region. The results were similar to the previous test, but with larger grain size and a lizard-skin feel to the texture. The glaze was mottled and less uniform. The smooth satin look was gone. I concluded one of the holds in the previous test hit the "sweet spot," at which point many small particles form. I did not know at which level.



test 6

Cool down: An uncontrolled drop to 2000°F, then -50° per hour to 1650°F.

Results: The slow cool from 2000°F to 1650°F gave a surface and color as in test 1, with a much greater number of gold particles. This also shows that the effects of test 4 depended on the 1650°F hold. This critical test showed that the greater color effect I wanted needed two holds.

Cool down: From Cone 6 to 2100°F at -50° per hour, then uncontrolled cooling to 1700°F, then -25° per hour to 1600°F.

Results: To test a second slow-cooling region, the kiln was cooled quickly from the top temperature to 1700°F, then slowly to 1600°F. The result was an intensely variegated effect with relatively few but larger particles in red and brown. The throwing lines were not prominent, so glaze thickness was not as important. The texture is lizard-skin satin, not the gloss of tests 1 and 5, nor the smooth satin of test 4. This result was related, but not quite like anything previous. This could be a jumping off point for a new series of tests.



test 7

a low end for controlled cooling, selecting intervals for markedly slow cooling in the temperature range 2200°–1450°F.

Speculation

With this limited series of tests, I produced a variety of textures and colors, by “poking” the cool-down profile. Each firing included

several identically glazed test pieces distributed throughout the kiln. I obtained an encouraging indication that the different results were caused by the cooling-down profiles and not extraneous effects. I next will explore whether maximal particle size growth takes place “hotter” than the temperature at which the greatest number of particles is formed. Cooling to ap-

proximately 1600°F, then reheating to around 1800°F should obtain both good numbers and development of microcrystals.

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