Question: Does sub-zero procedure change any characteristics of O1 Tool Steel?

Experiment: Compare four pieces of O1 tool steel for hardness (average of five measurements with rebound tester), flexibility and toughness using four different heat treating procedures.

Piece #1: Usual method—no change

1. Perform hardening in usual manner.
2. After quench, place in oven @400° F for 1 hour.
3. Rapidly cool and temper @ 650° F for 1 hour. HRC **54.0**

Piece #2: Usual method, but with Increased 2nd temper temperature to 750° F

1. Perform hardening in usual manner.
2. After quench, place in oven @400° F for 1 hour.
3. Rapidly cool and temper @ 750° F for 1 hour. HRC **51.9**

Piece #3: One hour temper @ 400° F and then <u>Sub-zero</u> treatment before final temper @ 750° F

1. Perform hardening in usual manner.
2. After quench, place in oven @400° F for 1 hour.
3. Remove from oven, rapidly cool to room temp.
4. Place in Kerosene/dry ice bath and leave in overnight
5. Remove from Kerosene/dry ice bath and allow piece to warm to room temp.
6. Temper @ 750° F for 1 hour. HRC **52.5**

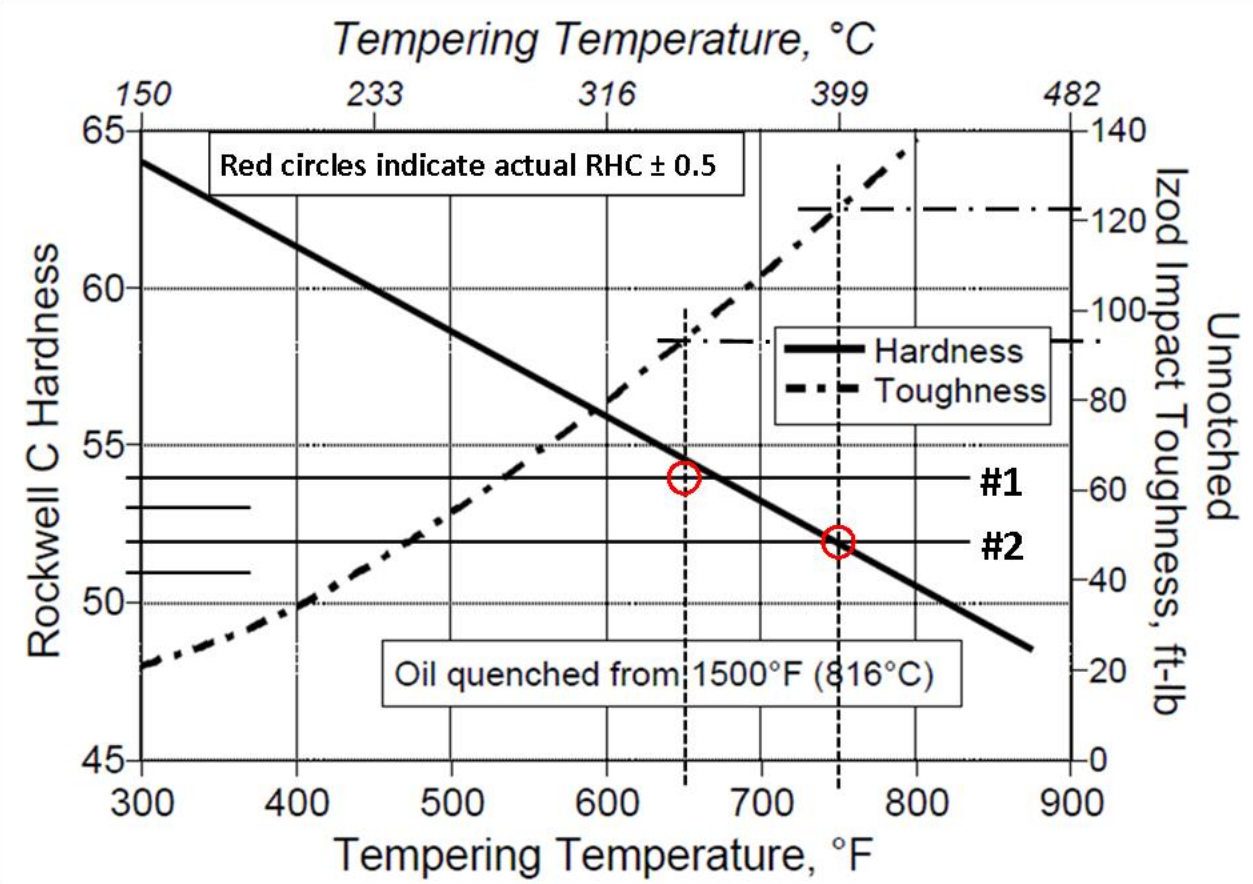
Piece #4: <u>Sub-zero</u> plunge directly after quench then temper @ 400° F before final temper @ 750° F

1. Perform hardening in usual manner.
2. After quench rapidly cool to room temp.
3. Place in dry Kerosene/dry ice bath and leave in overnight.
4. Remove from Kerosene/dry ice bath and allow piece to warm to room temp.
5. Place in oven @400° F for 1 hour.
6. Remove from oven, rapidly cool to room temp.
7. Temper @ 750° F for 1 hour. HRC **52.7**

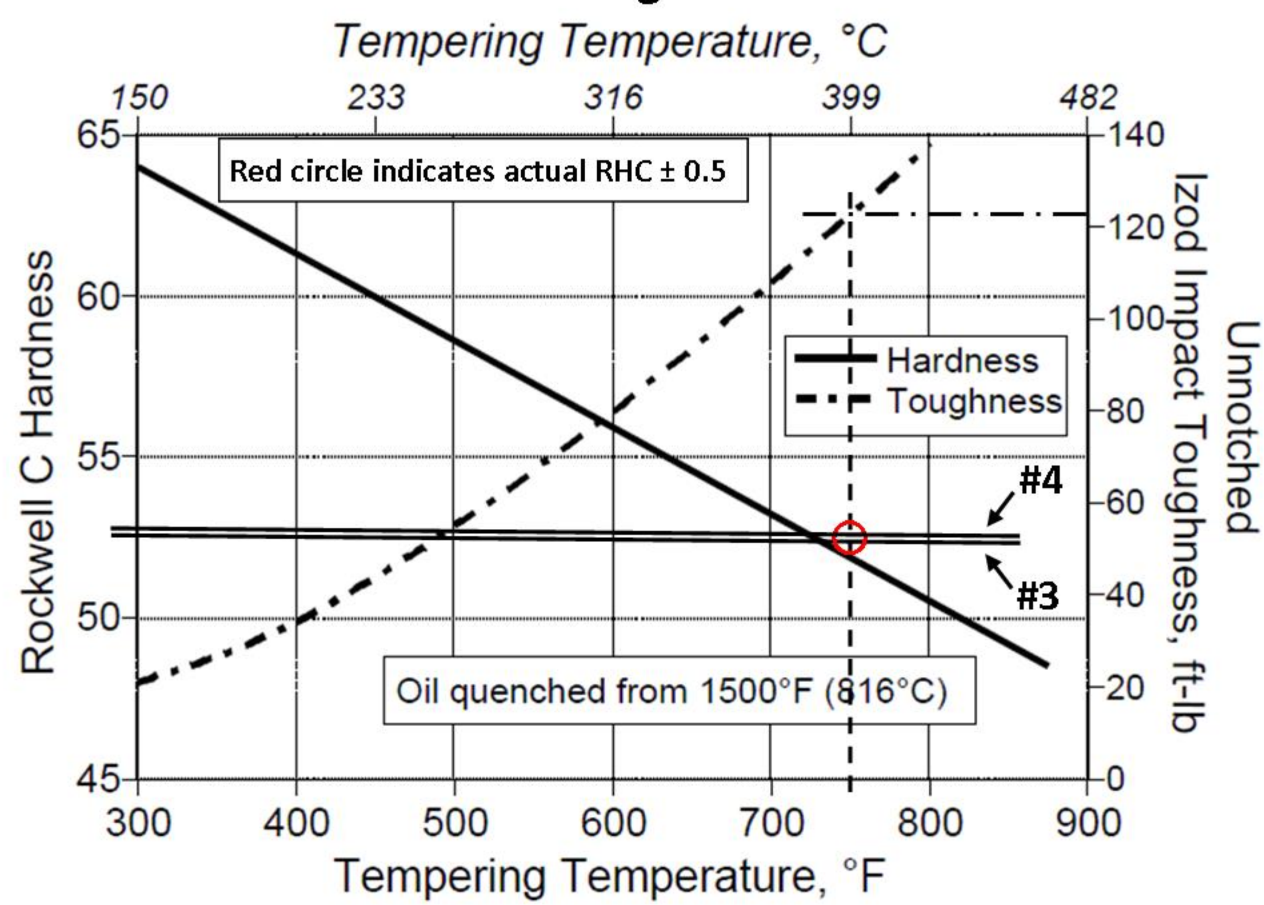
Hardness test results (refer to annotated Latrobe Specialty Steel charts):

- Piece #1 hardness matches expectation for 650° F temper, within ±0.5 HRC.
- Rapid cooling to room temperature after tempering seems to increase hardness relative to slow cooling by ~ 1 – 2 Rockwell units. I'll retest this later.
- Piece #2 hardness matches expectation for 750° F temper, within <0.5HRC.
- Pieces #3 and #4 (sub-zero treatment) are ~0.7 HRC harder than piece #2.
- Pieces #3 and #4 are virtually identical in hardness.
- Sub-zero treatment appeared to increase hardness by ~0.7 HRC

O1 Tool Steel Hardness vs. Toughness: Test Pieces #1 and #2



O1 Tool Steel Hardness vs. Toughness: Test Pieces #3 and #4



Details of Hardness test procedures

All pieces to be tested are 0.5” x 0.375” x 5”

Pieces # 1 - #4

1. Place in furnace at 500° F to prepare for decarb coating.
2. Heat for 10 minutes and coat.

Pieces #1 and #2 (process performed in the morning, separate from #3 and #4)

1. When coated, place back in furnace and set to 1200° F.
2. When temp is reached, hold for 10 minutes.
3. Set furnace to 1460° F.
4. When temp is reached, hold for 15 minutes.
5. While holding at temp place oil heater in oil (temp ~ 140° F).
6. After 15 min, quench both pieces.
7. Immediately place in oven at 400° F for 1 hour.
8. Remove both and plunge into water to bring to room temperature.
9. Bring furnace temp down to 650° F and place #1 in for 1 hour.
10. Remove and plunge into water to bring to room temperature.
11. Set furnace temp to 750° F and place #2 in for 1 Hour.
12. Remove and plunge into water to bring to room temperature.
13. Polish both pieces, test for hardness and record.

Pieces #3 and #4 (process performed in the afternoon, separate from #1 and #2)

1. Place coated pieces into oven set to 500° F (for consistency).
2. Set furnace to 1200° F.
3. When temp is reached, hold for 10 minutes.
4. Set furnace to 1460° F.
5. When temp is reached, hold for 15 minutes.
6. While holding at temp place oil heater in oil (temp ~ 140° F).
7. After 15 min, quench both pieces.
8. Immediately place #3 in oven at 400° F for 1 hour.
9. Place #4 in water to bring to room temp.
10. Place #4 directly into dry ice bath (leave overnight) when at room temp.
11. Remove #3 from oven and plunge into water to bring to room temperature.
12. Place #3 directly into dry ice bath (leave overnight) when room temp.
13. Allow #4 to warm to room temp and place in oven at 400° F for 1 hour.
14. Bring furnace temp to 750° F and place #3 and #4 in for 1 hour.
15. Remove and plunge into water to bring to room temperature.
16. Polish both pieces and test for hardness and record.

Testing for Flexibility, Breaking Point, of Sample Pieces

Pieces chosen for heat treat testing were left over from making throwing spikes. They were selected for thickness (0.375”) to maximize compressive-to-tensile stresses. The first tests performed were flexibility @ HRC.

Flexibility @ HRC was tested using a twenty ton press. The pieces were placed between two steel bars separated by four inches. A 1” dia. brass rod was placed at the center of the bar under test and downward pressure was applied until the test piece fractured. A dial gauge allowed for measurement of downward advance and fracture points.

Soft Reference Piece


Side View of Fractures

Some observations, so far:
As expected, hardness determined depth of fracture points. The fractures were spectacular (view video on No Spin RAT). Pieces #3 and #4 were sub-zero treated prior to final temper. Except for sub-zero treatment, pieces #2, #3 and #4 were heat treated the same: 750° F for one hour followed by rapid cool to room temp. It is reasonable to assume that the sub-zero

treatment is the reason for an increase in hardness of ~0.7 HRC for pieces #3 and #4 over piece #2. More investigation is needed.

Since three of these pieces, tempered at 750° F, had approximately the same fracture point, I feel confident with the statement that sub-zero treatment for "aging" before tempering, for the purpose of reducing Residual Austenite (RA), had no other observable effects beyond a slight increase in post-temper hardness of ~ 0.7 HRC due to reduced RA.

In future, I will be attempting to view grain structure, if possible, of each piece to determine if there is any difference between grain sizes based on sub-zero treatment. If I can, I plan to test an earlier made knife, yes, by fracturing it. In the early days of knife making, I always allowed knives to cool down naturally after tempering. So, some process variables to consider for further testing are, normal cooling to room temp, rapid cooling to room temp (placing in water), sub-zero cooling before temper, no sub-zero cooling before tempering.

Although I was originally considering S7 tool steel, due to its toughness, when I began this project, further research (refer to my "Making Tough No spin Knives" article for details) has made me realize that O1 tool steel is stated to be approximately four times tougher (~140 ft/lbs.) at HRC 51 than at HRC 60 (reference for toughness comparison). It is also more forgiving on the temper/toughness curve than S7.

My current plan for future knives is to continue using O1 tool steel, tempering at 800° F for the second temper, after sub-zero treatment before tempering and rapid cooling to room temp after tempering. This should yield an HRC of ~51 while providing for low RA and possible greater toughness than S7 tool steel at its toughness peak (~125 ft/lbs.) of HRC 57.

I say "possible" because it is really hard to compare Izod and Charpy. There is an Izod to Charpy conversion formula, Izod value x 2.738223 = Charpy. That's great, except the Izod test chart provides values for an <u>un-notched test</u> and the conversion formula is for a notched test. Still, an Izod value in ft/lbs. will convert to a larger Charpy value in ft/lbs.

Toughness will be the subject of future tests, using new pieces based on performance of first set of pieces. One end of a piece will be supported, a weighted pendulum will be released and strike at the highest point of the piece under test. This is essentially a modified (crude) Izod un-notched test. It will have no universal significance, but will provide a comparison among the samples. Stay tuned.