## SPRAY FORMED SUPERALLOY 625 PIPING

### A L. Moran\* and R.E. Rebis+

\*Department of Mechanical Engineering, United States Naval Academy
+Survivability, Structures, and Materials Directorate,
Carderock Division, Naval Surface Warfare Center
Annapolis MD 21402, USA

#### Abstract

Spray formed Alloy 625 piping subjected to standard amounts of cold reduction performed comparably to conventionally processed Alloy 625 piping and met the chemical analysis specifications and metallurgical requirements for porosity, oxide content and grain size required for Navy applications. Mechanical testing indicated that the tensile strength and ductility of roll extruded and pilger rolled pipes easily exceeded the minimum requirements while results of the hardness testing and Charpy impact testing showed no significant difference between as-sprayed preforms, spray formed and processed thin-walled piping, and conventionally produced piping. Results of fatigue testing of spray formed Alloy 625 pipes indicated that post processing is necessary to achieve the fatigue performance in terms of endurance limit of conventionally processed Alloy 625 pipes. Of the two post processing methods employed in this study, both methods diminished porosity and produced a fine grain size but roll-extrusion yielded a more uniform microstructure and better fatigue and strength properties. A weldability evaluation indicated that the hot-cracking resistance and mechanical properties of welded, spray formed Alloy 625 products were at least equivalent to that of conventional Alloy 625. Additionally, spray formed Alloy 625 piping performed comparably in service testing to wrought piping. Thus, Alloy 625 piping produced from spray formed tubulars appears to be a viable substitute for conventionally produced Alloy 625 piping.

#### Introduction

The Metals Department of the Carderock Division of the Naval Surface Warfare Center conducted an evaluation of spray formed Alloy 625 piping under the sponsorship of the Office of the Secretary of Defense Foreign Comparative Test Program. Spray forming, developed by Osprey Metals, Ltd in the United Kingdom, produces a characteristic fine equiaxed microstructure in a near net shape product [1]. The objective of the evaluation was to characterize the as-deposited spray formed Alloy 625 preforms and determine if these preforms could be processed into piping meeting current specification requirements and exhibiting equivalent or improved properties in comparison to conventionally produced piping. Three sizes of NPS (Nominal Pipe Size) Class 850 Alloy 625 pipe (10.2, 20.3 and 35.6 cm), produced to Electric Boat specification 3200C on Pipe and Tube, Seamless, Nickel-Chromium-Molybdenum Alloy Procurement, date 1989, were tested as

representative of the range of sizes producible by the spray forming technique. In order to obtain material for testing, a contract was awarded to Sandvik Steel in Sandviken, Sweden to produce Alloy 625 as-sprayed preforms and finished piping via the Osprey spray forming process. [2] Four as-sprayed tubular preform sections were provided for evaluation. Additional as-sprayed preforms were cold worked and reduced to the final EB 3200C specification dimensions by either pilger rolling at Sandvik or roll extrusion at Kaiser Rollmet in Irvine, California. Since the pilger rolling process is typically limited to tubulars less than 25.4 cm in diameter, roll extrusion was required on the larger diameter piping.

# **Experimental Results**

NSWC-CD conducted testing to verify the chemical, dimensional, metallurgical and mechanical properties of the Alloy 625 spray formed material.

# Chemical and Microstructural Analysis

Chemical analyses of the spray formed preforms and finished thin-walled piping were performed in accordance with ASTM E38, E354 and Fed-STD-151 method 112. All of the Alloy 625 spray formed preforms and piping met the chemical analysis specifications required in Electric Boat 3200C. Differences between piping derived from spray formed tubulars and conventional ingot castings appeared to be the level of carbon and nitrogen in each alloy. The spray formed product was generally lower in carbon and higher in nitrogen. Both the carbon and the nitrogen contribute to the formation of carbonitrides in the alloy. For the preforms, the average porosity was less than 0.04%, well below the accepted 2% porosity level, with the pores being very fine in size, evenly shaped and discontinuous. The range of average grain size was between 30 to 45 microns, well below the required average grain size of less than 100 microns. The 10.2 cm spray formed/pilger rolled piping had an average grain size of 14 microns and several carbide bands, which are formed during the pilger rolling process. Although the grain sizes of the roll extruded and pilger rolled materials were similar, the spray formed/roll extruded microstructure appeared to be more uniform than the spray formed/pilger rolled microstructure. The average grain size for the pilger rolled as well as the roll extruded piping was between to 10-15 microns. The porosities for all the finished piping were less than 0.03% for the roll extruded and 0.02% for the pilger rolled materials.

### Non-destructive Evaluation

Penetrant testing (PT) of the 10.2 cm pilger rolled piping was conducted in accordance with MIL-STD-271 on a 24.5 m length of piping from twenty pipe sections. The inspected area was 100%. In accordance with NAVSEA 9000-LP-003-8000, any discontinuity exceeding 0.16 cm was considered a rejectable indication. Nine indications were detected that exceeded the criteria. Seven of nine indications were 0.41 cm in length or less. The results of the penetrant testing (100%) of both 20.3 cm roll extruded and 20.3 cm pilger rolled piping noted 67 indications detected over 4.4 m of pipe, while the pilger rolled material had 21 indications detected over 10.2 m of pipe. For the 35.6 cm piping, the inspected area ranged from 90 to 100%. A total of 10 indications were found over the 24 m of 35.6 cm piping inspected and 9 out of the 10 indications were less than 0.64 cm long. Surface indications that exceed the rejectable criteria can be ground out if the following requirements are satisfied.

The results of the ultrasonic testing (UT), completed according to MIL-STD-271F, of the 10.2 cm pilger rolled, 20.3 cm pilger rolled, 20.3 cm rolled extruded piping indicated that no rejectable

indications were detected. In addition, no rejectable indications were detected on the as-sprayed and machined preforms. Ultrasonic testing of the 35.6 cm piping was performed on approximately 80% of the pipe area. Rejectable indications were detected in three pipes. All of the rejectable indications exceeded 100% full screen height (FSH). One pipe had two rejectable indications, one measuring 0.28 cm deep, the other 0.53 cm deep. The second pipe also had two rejectable indications, one measuring 0.46 cm deep, the other 0.30 cm deep. The third pipe had one rejectable indication measuring 0.36 cm deep.

## Mechanical Testing

The tensile properties for the finished thin-walled piping and as-sprayed preforms are listed in Table I. The yield stresses of the roll extruded and pilger rolled pipes exceeded the 379 MPa minimum requirement. The roll extruded pipes (20.3 cm and 35.6 cm) and the conventional pipe well exceeded the minimum yield stress, while the yield stresses of the pilger-rolled pipes were lower. The difference is attributed to different reduction/annealing cycles used by the different manufacturers. The pilger-rolled, roll extruded and conventionally processed pipes all exhibited similar ultimate stresses ranging from 940 to 968 MPa, which exceeded the 758 MPa minimum requirement. The 20.3 cm pilger-rolled pipe showed a slightly lower ultimate stress of 896 MPa. The percent elongation for all the pipes met the minimum 30%, ranging from 38 to 41%. These values were 10 to 20% lower than the preforms. This reduction was expected due to the strain-hardening induced during the pipe finishing process. The yield stress of the as-sprayed preforms were all below the specified minimum value for reduced piping, while the ultimate stress exceeded the minimum requirement. The elongation values of the preforms also exceeded the minimum value of 30%. The values ranged from 49 to 59%. These lower strength, higher ductility as-sprayed preforms were intentionally produced to facilitate workability during pilger rolling and roll extruding.

Average Brinell hardness measurements for the as-sprayed preforms were between 183 and 197 for both outer and inner diameters. Corresponding Rockwell hardness measurements ranged from RHB 87 to RHB 94. The average hardness was 95 RHB for the 10.2 cm pipe, RHB 96 for the 20.3 cm pilger-rolled pipe, and RHB 99 for the 35.6 cm pipe. Average hardness levels of the 20.3 cm roll-extruded pipe and conventional pipe were 23 and 24 RHC, respectively.

Results of the Charpy impact toughness tests are shown in Table II. Comparisons can only be made among specimens of equivalent size. The room temperature impact energies for the 0.32 cm subsize specimens ranged form 22 to 27 J for the preforms and from 14 to 23 J for the finished pipes. At these small energy levels it is difficult to assess whether the difference in energy is significant. The specimens that exhibited the lower energy levels came from two different preforms and were processed differently. Therefore, there does not appear to be a correlation to the source of the material. At -73°C the energy levels for the 20.3 cm conventional and 35.6 cm roll-extruded pipes averaged 20 J, and the 20.3 cm pilger-rolled and roll-extruded pipes averaged 11 J.

The fatigue performance in air of as-deposited spray formed Alloy 625 preforms compared to that of conventionally processed Alloy 625 is shown in Figure 1. This testing was conducted to determine a baseline fatigue curve for the as-sprayed product prior to cold-working by the roll extrusion and pilgering processes. The data includes three different as-sprayed preforms that were subsequently processed into 10.2 cm, 20.3 cm and 35.6 cm nominal diameter pipes. As expected, the stress vs. number of cycles (S-N) curves indicate that the fatigue performance of the as-sprayed preforms is inferior to the fatigue performance of the specimens from conventionally processed pipe when tested in air. However, after post processing of the spray formed preforms by means of roll-extrusion, the fatigue performance in air is comparable to that of the conventionally processed material, as shown

Table I. Strength properties for spray formed Alloy 625 preforms and piping.

MATERIAL	0.2% YIELD STRESS (MPa)Ave. of two tests	ULTIMATE TENSILE STRENGTH (MPa)	PERCENT ELONGATION
10.2 CM PREFORM	361	785	56
20.3 CM PREFORM	371	789	59
20.3 CM PREFORM	330	773	49
35.6 CM PREFORM	360	782	57
10.2 CM PILGER PIPE	459	954	42
20.3 CM PILGER PIPE	480	896	42
20.3 CM ROLLED PIPE	595	962	38
20.3 CM ROLLED PIPE	568	940	41
20.3 CM CONV. MFG.	561	968	41
35.6 CM ROLLED PIPE	561	953	41
EB3200C SPEC	379	758	30

Table II. Charpy V-notch impact toughness of spray formed preform and Alloy 625 pipe.

MATERIAL	3 TEST AVE. ENERGY AT RT (J)	AVE.ENERGY AT -73C (J)
FULL SIZE 1.00CM SAMPLES 10.2 CM PREFORM	115	-
20.3 CM PREFORM	96	-
SUBSIZE 0.76CM SAMPLES 20.3 CM PREFORM	73	-
35.6 CM PREFORM	68	_
SUBSIZE 0.32CM SAMPLES 10.2 CM PREFORM	23	<u>-</u>
20.3 CM PREFORM	23	-
20.3 CM PREFORM	27	-
35.6 CM PREFORM	22	<u>-</u>
20.3 CM PILGERED PIPE	14	11
20.3 CM EXTRUDED PIPE	14	11
20.3 CM EXTRUDED PIPE	22	-
20.3 CM CONV MFG PIPE	23	22
35.6 CM EXTRUDED PIPE	20	20
SUBSIZE 0.27CM SAMPLES 10.2 CM PILGERED PIPE	19	-
35.6 CM EXTRUDED PIPE	16	

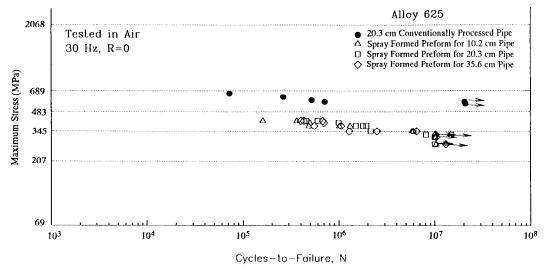


Figure 1. S-N curve for spray formed Alloy 625 pipe preforms tested in air.

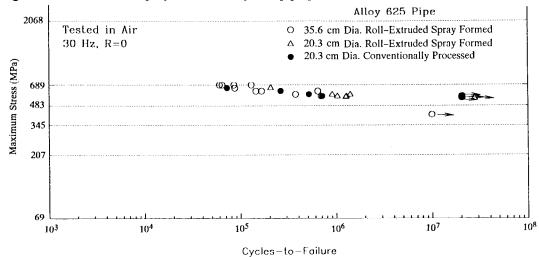


Figure 2. S-N curve for the spray formed Alloy 625 roll-extruded and conventionally processed pipe tested in air.

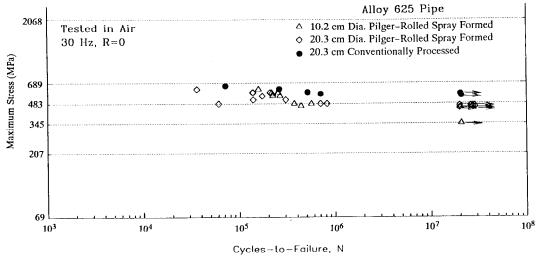


Figure 3. S-N curve for the spray formed Alloy 625 pilger-rolled and conventionally processed pipe tested in air.

in Figure 2. Figure 3 shows that post processing of the spray formed preforms by means of pilger-rolling improves fatigue performance in air compared to that of the preforms, but not to the same level as the conventionally processed and spray formed roll-extruded material. The fatigue performance of the roll-extruded spray formed and pilger-rolled spray formed Alloy 625 piping compared to that of the conventionally processed Alloy 625 piping when tested in 3.5% NaCl seawater is shown in Figures 4 and 5, respectively. The fatigue data in seawater of both spray formed (and subsequently processed) materials seems to compare well with the data of the conventionally processed Alloy. The run out stresses for the spray formed materials were slightly below that of the conventionally processed Alloy, but may be considered to lie within the scatter of the data.

The results of fatigue tests in air and seawater were performed on specimens from weldments made in a roll extruded spray formed Alloy 625 pipe and conventionally processed Alloy 625 pipe using the GTAW process. These specimens were tested with the weld reinforcement intact. This resulted in a reduced fatigue performance due to this notch factor. The S-N curves indicated that the weldments in the spray formed roll-extruded pipe performed as well as weldments in conventionally processed pipe in both environments. The fatigue strengths of specimens from GTAW weldments of both the roll-extruded spray formed pipe and the conventionally processed pipe were slightly lower in the seawater environment compared to the data collected in air.

Fatigue data was also gathered from testing specimens from roll-extruded spray formed Alloy 625 pipe and conventionally processed Alloy 625 pipe, welded using the GTAW root, SMAW fill procedure, in air and seawater environments. In each environment, the spray formed roll-extruded pipe weldment data was within the scatter of the conventionally processed pipe weldment data. The seawater environment seemed to have a slightly negative effect on both the roll-extruded spray formed and conventionally processed Alloy 625 weldments, but to an equal extent.

#### Weldability Assessment

A number of methods have been applied to estimate and compare hot cracking susceptibilities from hot ductility test data. Recently, Lippold et al. [3] developed a methodology that uses melting temperature, zero ductility temperature, zero strength temperature, and ductility recovery temperature to evaluate cracking susceptibility. Their method uses these four parameters to define a thermal crack susceptible region. The thermal crack susceptible region represents a heat affected zone region surrounding the weld pool within which the material has no ductility and is thus susceptible to hot cracking. The thermal crack susceptible region has an on-heating component and an on-cooling component. These components account for the possibility of initiating a heat affected zone hot crack during weld heating or cooling (i.e., at locations ahead of or behind the instantaneous weld center). The on-heating component of the thermal crack susceptible region, termed the on-heating crack susceptible region, is defined as the difference between melting temperature and the zero ductility temperature. The higher the value for this value, the greater the susceptibility to hot cracking during the heating cycle. The on-cooling component of the crack susceptible region, known as the brittle temperature range, is defined as the difference between the peak temperature used in the on-cooling phase and the ductility recovery temperature. The brittle temperature range is a function of peak temperature and represents the range that has no ductility during the cooling portion of the weld. Increasing brittle temperature range values indicate higher susceptibility to hot cracking. Table III presents the extent of the thermal crack susceptible region for the spray formed alloy 625 preforms as compared to data reported for conventional alloy 625. The data show that the crack susceptible region of spray formed alloy 625 compares well to the conventional alloy 625 crack susceptible region.

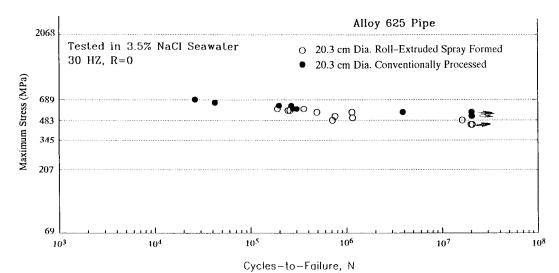


Figure 4. S-N curve for the spray formed Alloy 625 roll-extruded and conventionally processed pipe tested in seawater.

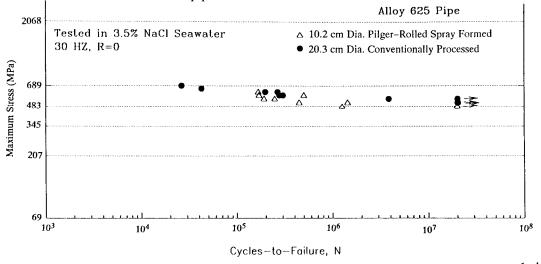


Figure 5. S-N curve for the spray formed Alloy 625 pilger-rolled and conventionally processed pipe tested in seawater.

Table III. Thermal crack susceptible regions of spray formed and conventional Alloy 625.

MATERIAL	SPRAY FORMED 625 PREFORMS	CONVENTIONAL 625 (LIPPOLD ET AL, 1992)
APP. MELTING TEMP, T <sub>L</sub> (°C)	1349	1322
ZERO-DUCTILITY TEMP, ZDT (°C)	1260-1302	1220
ZERO-STRENGTH TEMPERATURE, ZST (°C)	1343-1349	1313
DUCTILITY RECOVERY TEMPERATURE, DRT <sub>ZST</sub> (°C)	1154-1182	1149
ON HEATING CRACK SUSCEPTIBLE REGION, CSR ( $T_L$ -ZDT)	29-71	84
ON COOLING BRITTLE TEMPERATURE RANGE, BTR <sub>ZST</sub> (ZST-DRT)	143-171	146

The specification requirements and tensile results from test weldments in spray formed alloy 625 and conventional alloy 625 piping indicated that the weldment tensile strengths exceeded the specification requirement, ranging from 773 to 890 MPa. In general, the weldment tensile strengths were below the tensile strength of the pipe. Additionally, all weldment specimens fractured in the weld metal, also indicating that the weld metal tensile strength was less than that of the pipe. Table IV presents the CVN results from the simulated-HAZ specimens. Results from the as-sprayed preforms are included for comparison. The data show that the HAZ and as-sprayed materials displayed comparable CVN properties.

## Corrosion Testing

Crevice corrosion testing was conducted on 20.3 cm diameter conventionally manufactured Alloy 625 piping, spray formed/roll extruded piping, and spray formed/pilger rolled piping. This testing was conducted for 60 days in 30°C filtered seawater. For the conventionally produced Alloy 625 piping, eight sites out of 156 sites (5%) were attacked. The spray formed/roll extruded 20.3 cm diameter piping was immune to crevice corrosion on all of the 132 sites tested. For the spray formed/pilger rolled 20.3 cm diameter piping, 11 sites out of 68 sites (16%) were attacked. Based on this data, it appears that the crevice corrosion susceptibility of the piping manufactured from spray formed preforms via roll extruding is comparable to that of the conventionally produced material. Although the spray formed/pilger rolled material showed a slightly higher incidence of attack and slightly more active corrosion potentials during testing, this data was not considered sufficient to conclude that this material was significantly more susceptible to crevice attack than the spray formed/roll extruded or conventionally produced materials.

Crevice corrosion testing of GTA and SMA welded 20.3 cm diameter spray form/roll extruded, spray form/pilger rolled and conventional Alloy 625 piping was also conducted for 60 days in 30°C filtered, seawater. The results were that 4 of the 16 welded crevice sites on the conventional piping were attacked, 1 of 16 welded crevice sites on the spray form/roll extruded piping were attacked, and 1 of 16 welded crevice sites on the spray formed/pilger rolled piping were attacked. This incidence of attack (6 out of 48 sites, 12.5%) is approximately twice that observed for the base metal (19 sites out of 356, 5.3%) indicating that crevices formed on welds in Alloy 625 should be more prone to crevice corrosion than those formed on base materials. Of the six weld metal sites which corroded, four of the sites were GTA welds and two were SMA welds. In light of the low incidence of attack, it is not believed that this result indicates that GTA welds are more susceptible to crevice corrosion than SMA welds. In most cases, preferential attack in the fusion zone/heat affected zone was observed.

The results of the base metal and weldment testing suggest that the spray formed/roll extruded piping has crevice corrosion performance that is equivalent to the conventionally produced material. The spray formed/pilger rolled material showed a slightly higher incidence of attack. However, this data was not considered sufficient to conclude that this material was significantly more susceptible to crevice attack than the spray formed/roll extruded or conventionally produced materials.

The results of stress corrosion cracking evaluation via slow strain rate testing included the breaking loads at various potentials divided by the breaking loads in air. This allows for comparisons between the three product forms and mitigates the impact of wall thickness variability between the product forms. The results indicated that no significant reduction in breaking load occurs at open circuit conditions or -0.85 mV vs. SCE, which is the standard target potential for Impressed Current Cathodic Protection (ICCP) systems on Navy ships. At -1.00V vs. SCE, which is the potential of zinc or aluminum sacrificial anode cathodic protection systems, the roll extruded and pilger rolling

spray formed piping experienced an average of 11% reduction in maximum load compared to specimens tested in air. The conventionally processed piping exhibited the smallest average reduction in maximum load (5%) at -1.0 mV vs. SCE. Specimens tested at very negative potentials, -1.250 V vs. SCE, were significantly less resistant to hydrogen assisted cracking. All specimens tested experienced sharp reductions in maximum load as compared to the same materials tested in air. Both the pilger rolled and roll extruded piping tested at -1.25V vs. SCE experienced the greatest reduction in average maximum loads (29% and 33%, respectively). The average maximum load of the conventionally processed piping, although less severely affected than the spray formed based material, was 16% less than the average maximum load for specimens tested in air. Significant reduction in the load bearing capability of any of the three test materials did not occur until specimens were tested at very negative potentials (-1.250V vs. SCE). This potential is significantly more negative than would be seen in service with zinc or aluminum sacrificial anode cathodic protection. Testing at lower potentials increases the hydrogen overpotential and the concentration of hydrogen available to diffuse into the test specimen. Consequently, the environment at this potential is likely to be more aggressive than the worst case scenario envisioned in shipboard service. Testing performed under freely corroding conditions most closely resembles the expected shipboard environment for the majority of a sea water piping system. Typically a sea water piping system is not cathodically protected with the exception of piping ends located in the proximity of anodes or in electrical contact with less noble materials. In service the sea water piping potential would be expected in the range of -90 to -120 mV, the open circuit potential of Alloy 625 in sea water. Under freely corroding conditions, the performance of all three materials was comparable to corresponding specimens tested in air. No embrittlement was evident at potentials expected for sea water piping service.

### **Conclusions**

A minimal amount of cold working is required on the as-sprayed Alloy 625 preforms for them to meet the requirements of the EB 3200C specification. Spray formed Alloy 625 piping subjected to standard amounts of cold reduction performed comparably to the conventionally processed Alloy 625 piping and met the chemical analysis specifications and metallurgical requirements for porosity, oxide content and grain size required for Navy applications. The tensile strength and ductility of all the roll extruded and pilger rolled pipes easily exceeded the minimum requirements while results of the hardness testing and Charpy impact testing showed no significant difference between the as-sprayed preforms, the spray formed and processed thin-walled piping, and the conventionally produced piping. Results of the fatigue testing of the spray formed Alloy 625 pipes indicate that post processing is necessary to achieve the fatigue performance in terms of endurance limit of conventionally processed Alloy 625 pipes. Of the two post processing methods employed in this study, both methods diminished porosity and produced a fine grain size but roll-extrusion yielded a more uniform microstructure and the better fatigue and strength properties. The weldability evaluation indicated that the hot-cracking resistance and mechanical properties of the welded, spray formed Alloy 625 products were at least equivalent to that of conventional Alloy 625. Additionally, spray formed Alloy 625 piping performed comparably in corrosion testing to wrought piping. Thus, Alloy 625 piping produced from spray formed tubulars appears to be a viable substitute for conventionally produced Alloy 625 piping.

Table IV. Room temp Charpy V-notch results for Alloy 625 simulated HAZ specimens.

MATERIAL	SPECIMEN TYPE	ABSORBED ENERGY (J)
20.3 CM PREFORM	HAZ	27, 28, 30, 20, 27 27 AVERAGE
20.3 CM PREFORM	HAZ	38, 37, 30, 35, 37 35 AVERAGE
20.3 CM PREFORM	BASE METAL	23 AVERAGE
20.3 CM PREFORM	BASE METAL	27 AVERAGE

All specimens were subsize 0.318 x 1.00 cm

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