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CROSS-VALIDATION OF THERMAL EXPANSION MEASUREMENTS

STAN SMITH, PETRA MILDEOVA, ROGER MORRELL

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Materials and Mechanical Metrology

Advanced Engineering Materials

ABSTRACT

Thermal expansion, usually expressed as the coefficient of thermal expansion, is an important thermophysical property and there is a need to obtain highly reliable values. The aim of this cross-validation work was to compare and validate a newly acquired mechanical dilatometer (Linseis L75VS) to existing mechanical dilatometers and a thermomechanical analyser.

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National Physical Laboratory

Hampton Road, Teddington, Middlesex, TW11 0LW

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Approved on behalf of NPLML by
Stefanos Giannis, Science Area Leader, Advanced Engineering Materials

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1. INTRODUCTION

Thermal expansion, usually expressed as the coefficient of thermal expansion, is an important material property and there is a need to obtain highly reliable values for engineering purposes. NPL has recently acquired a new single push-rod mechanical dilatometer (Linseis L75VS) for the measurement of thermal expansion between nominally -150 °C and 500 °C to replace an existing older modified mechanical dilatometer that has been used mainly for measurements around or below room temperature. The aim of this work was to compare and validate the new instrument against existing instruments – a low-temperature modified Linseis dilatometer, a high-temperature Linseis L75 dilatometer and a TA Instruments thermo-mechanical analyser (TMA).

The mechanical dilatometers are operated according to ASTM E228-22 and the thermomechanical analyser according to ASTM E831-19.

The test-piece materials used in this work were copper, molybdenum, platinum, stainless steel 316L, Inconel 718 and Pyroceram 9606, which all have good supporting literature and/or reference data from NIST or other sources. The test-pieces of each material were measured between -100 °C and 800 °C according to the following matrix.

Instrument	Temperature range of tests	Materials measured					
		Copper	Platinum	Pyroceram 9606	Stainless steel 316L	Inconel 718	Molybdenum
Modified Linseis Gen 2	-50 to 190 °C	✓	-	✓	-	✓	✓
Linseis L75	20 to 800 °C	✓	✓	✓	✓	✓	✓
Linseis L75VS	-100 to 500 °C	✓	✓	✓	✓	✓	✓
TMA	-50 to 300 °C	✓	✓	✓	✓	-	✓

It was first attempted to run the new Linseis L75VS from the minimum specified temperature of -150 °C but the dewar supplied with the instrument was too small to be able to reach that temperature at the conventionally used slow heating/cooling rate. A larger dewar was later employed but also proved insufficient. It was therefore decided to perform the comparison by operating the instrument only to -100 °C instead.

2. MEASUREMENT METHODS

All four instruments were operated in a similar manner using a heating and cooling rate of 1, 2 or 3 °C/min over three or four heating and cooling cycles, using the above-described calibration routines, and data analysed and compared on similar bases. Note that stepwise temperature ramping is rarely used as being uneconomical.

The output data in terms of displacement in micrometres as a function of temperature were processed manually for each thermal cycle using a spreadsheet method, and then converted to fractional length change in parts per million and mean expansion coefficient from 25 °C. This procedure is independent of the equipment software and follows the procedure in ASTM E228-22.

2.1 LINSEIS L75 MECHANICAL DILATOMETER (RT TO 1500 °C)

The Linseis L75 is a horizontal alumina twin push-rod mechanical dilatometer designed to be operated from room temperature to 1500 °C. Two test-pieces are run in parallel, normally at 2 °C/min heating and cooling rate. The usual operating procedure is to run a baseline for both test-piece stations using alumina test-pieces, and then the test-piece(s) are run over three heating and cooling cycles over an identical temperature profile. Test-pieces are usually 5 mm diameter x 30 mm long rods or 5 x 5 x 30 mm bars with flat smooth ends square to length. Calibration of the transducer system is set by the manufacturer and the overall behaviour regularly checked against a platinum reference.

2.2 MODIFIED LINSEIS GEN 2 MECHANICAL DILATOMETER (-100 °C TO 200 °C)

This is an old low-temperature vertical silica single push rod mechanical dilatometer designed to operate between -100 °C and 200 °C. It was built in 1970s and has a large flat base to the test-piece holder which can accommodate large test-pieces of up to 40 x 40 x 5 mm, which makes it ideal for the measurement of composite materials where the test-piece must be large enough to represent the bulk material, such as composite panel materials. Thermal cycles consist of controlled cooling below room-temperature, then controlled heating to the maximum required temperature, followed by controlled cooling to about 50 °C after which the system cools naturally to room temperature. Four thermal cycles are commonly made at a nominal heating/cooling rate of 1 °C/min or slower for larger test-pieces. Below room temperature measurements are using a 120-litre unpressurised liquid nitrogen dewar. Calibration of the transducer is made using a calibrated drum micrometre and the overall behaviour is checked using a copper reference.

2.3 LINSEIS L75VS MECHANICAL DILATOMETER (-150 C TO 500 °C)

The Linseis L75VS is a vertical silica single push-rod mechanical dilatometer designed to operate between -150 °C and 500 °C. It is connected to a 60-litre liquid nitrogen dewar to allow measurements down to -150 °C. The system can accommodate large test-pieces up to about 40 x 40 x 10 mm. It is operated at 3 °C/min on heating and cooling over three thermal cycles. The transducer system is calibrated by the manufacturer. Baseline and test-piece runs are used.

2.4 THERMOMECHANICAL ANALYSIS (-80 °C TO 800 °C)

The TA Instruments Thermomechanical Analyser Q400 is designed to operate between -80 °C and 800 °C, using silica apparatus. It is designed principally for use with high-expansion polymeric materials but was included because it has a sub-zero temperature capability. The 10 mm long test-pieces were measured in expansion mode using an expansion probe at 3 °C/min on heating and cooling. A refrigerator unit was used for cooling to -80 °C. New calibration procedures have been trialled including baseline determination and use of reference materials.

3. RESULTS

The results on each of the reference materials are dealt with separately in the following sections. They are represented in two ways: as fractional length change (ppm) as a function of temperature and as mean (or chord) expansion coefficient as a function of temperature range from a starting point (usually 20 or 25 °C).

Fractional length change gives the overall trend, and clearly demonstrates any offsets or phase changes during heating and cooling. Mean expansion coefficient is a useful engineering method of describing how a material expands. The numerical data obtained in this work are given in Tables 1 to 27. The main goal of the work to validate the new system in terms of the difference between the literature values and Linseis L75VS measured values are summarised in Table 28 and 29.

In this work, we have not evaluated ‘expansivity’, or tangent expansion coefficient, because that is less useful for engineering analysis and does not link directly to overall expansion behaviour.

3.1 COPPER

High-purity copper has long been used as a standard reference material. The source of the test material used was Goodfellow and is of 99.999% purity.

The results for the individual instruments are given in Figures 1 to 4 and are tabulated in Tables 1 to 4. As can be seen from the fractional length change plot in Figure 3, the new Linseis L75VS dilatometer produces slight thermal hysteresis, and this also occurs in the set of results for the old Linseis system. The result is repeatable over two heating and cooling cycles 1.

Overall, there is a good agreement between the four instruments, as can be seen from Figure 5 with only a slight difference in fractional length change between the reference data and measured values for the new dilatometer.

In Figure 6 a comparison is made between the mean expansion coefficients determined from a 25 °C origin. This exposes significant differences over small temperature ranges, with the TMA showing the greatest deviation from the reference data.

Table 5 gives an overall summary of the averaged data for each instrument.

[1] A possible reason for the apparent hysteresis being not readily removed by calibration routines is due to small temperature differences between the temperature-measuring thermocouple and the actual test-piece temperature as a result of differences in thermal mass and thermal conductivity of, and apparatus internal heat transfer to, the reference test-piece compared with baseline test-piece.

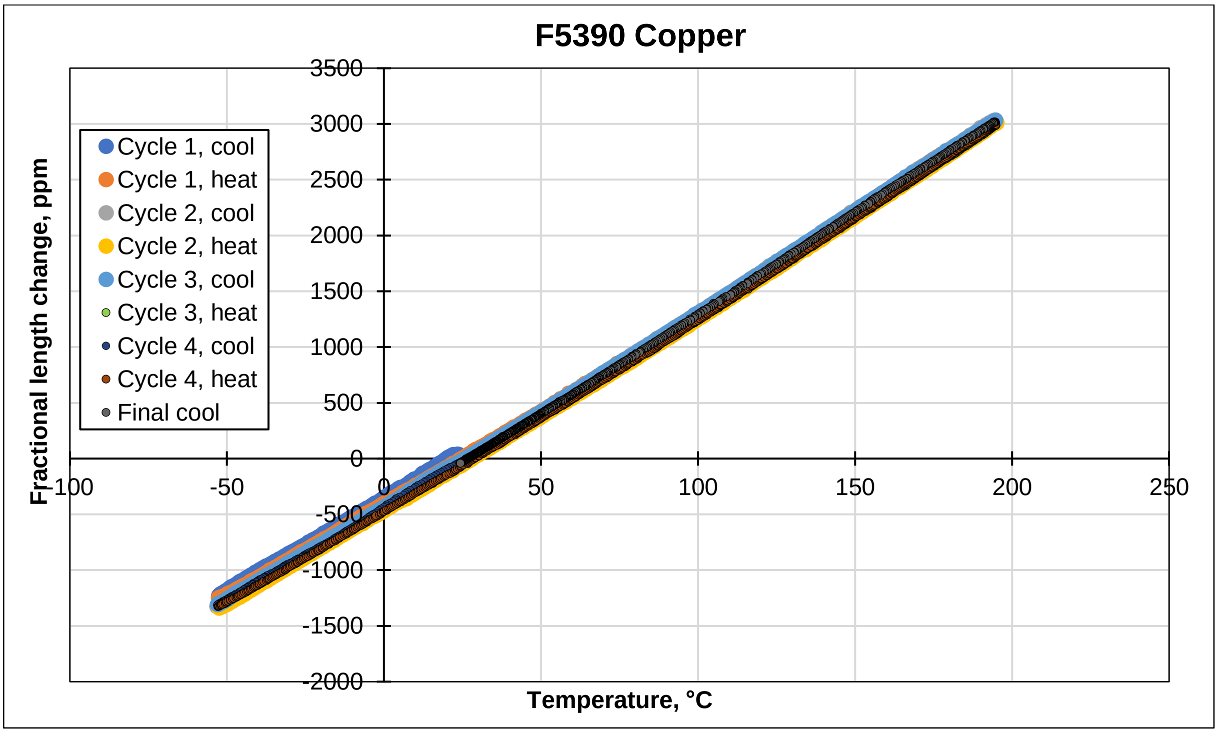


Figure 1: Modified Linseis Gen 2: Fractional length change as a function of temperature for a copper sample over four heating and cooling cycles

Table 1: Modified Linseis Gen 2: Fractional length change and mean expansion coefficient as a function of temperature

Temp, °C	Fractional length change, ppm, from 20 °C											Mean expansion coeff. from 25 °C, 10^{-6} K^{-1}	Std.dev.
	Cycle 1		Cycle 2		Cycle 3		Cycle 4		Final Cool	Mean from 25 °C	Std.dev.		
	Cool	Heat	Cool	Heat	Cool	Heat	Cool	Heat					
-50	-1220	-1221	-1252	-1251	-1244	-1228	-1241	-1225	-	-1235	13	16.5	-0.2
-40	-1047	-1070	-1085	-1083	-1077	-1078	-1071	-1069	-	-1073	12	16.5	-0.2
-20	-729	-740	-745	-744	-749	-753	-762	-747	-	-746	10	16.6	-0.2
0	-392	-497	-417	-413	-418	-416	-414	-413	-	-423	31	16.9	-1.2
20	-41	-83	-78	-79	-72	-88	-72	-85	-88	-76	15	15.2	-2.9
25	0	0	0	0	0	0	0	0	0	0	0	-	-
40	-	252	259	259	258	248	251	252	258	255	4	17.0	0.3
60	-	594	618	602	607	592	594	595	603	601	9	17.2	0.3
80	-	939	972	948	961	941	942	945	958	951	12	17.3	0.2
100	-	1288	1327	1308	1319	1292	1301	1302	1319	1307	14	17.4	0.2
120	-	1649	1691	1669	1680	1654	1665	1666	1683	1670	14	17.6	0.2
140	-	2018	2063	1973	2053	2023	2036	2032	2055	2032	29	17.7	0.2
160	-	2387	2430	2407	2422	2390	2404	2404	2428	2409	16	17.8	0.1
180	-	2764	2796	2779	2784	2759	2767	2776	2790	2777	13	17.9	0.1
190	-	2930	2977	2965	2967	2946	2949	2963	2969	2958	15	17.9	0.1

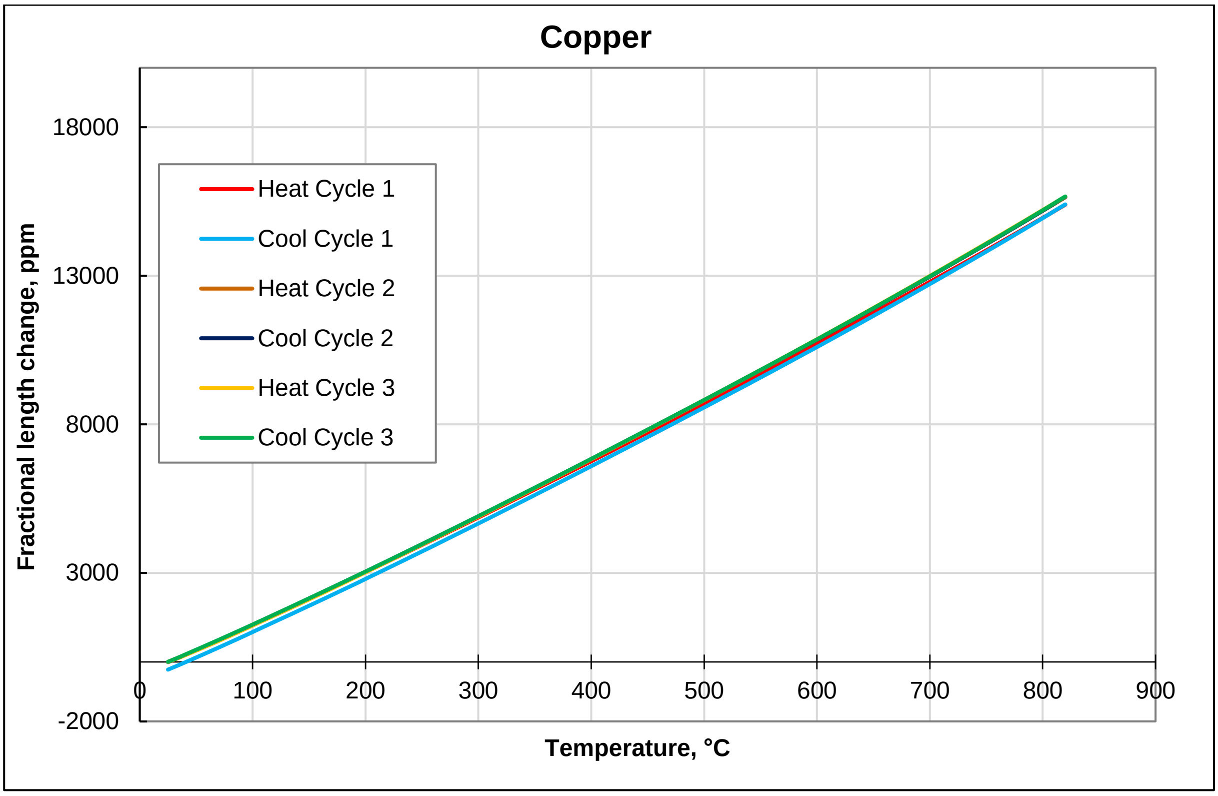


Figure 2: Linseis L75 high-temperature dilatometer: Fractional length change results for a copper sample using the high-temperature Linseis L75 dilatometer over three heating and cooling cycles.

Table 2: Linseis L75 high-temperature dilatometer: Fractional length change and mean expansion coefficient from Linseis high- temperature dilatometer

Temp., °C	Fractional length change from 25 °C, ppm								Expansion coeff from 25 °C, 10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev
50	396	407	386	405	385	406	396	12	15.8	0.5
100	1250	1265	1230	1256	1229	1254	1242	14	16.6	0.2
150	2125	2149	2111	2141	2107	2137	2124	17	17.0	0.1
200	3023	3051	3025	3043	3018	3036	3030	11	17.3	0.1
250	3936	3976	3953	3970	3948	3959	3957	10	17.6	0.0
300	4864	4918	4895	4908	4892	4903	4900	7	17.8	0.0
350	5807	5876	5853	5865	5851	5860	5857	7	18.0	0.0
400	6756	6846	6824	6838	6824	6830	6829	6	18.2	0.0
450	7723	7829	7810	7822	7812	7816	7815	5	18.4	0.0
500	8703	8824	8807	8818	8813	8813	8813	4	18.6	0.0
550	9702	9833	9818	9829	9827	9825	9825	5	18.7	0.0
600	10715	10858	10848	10856	10861	10853	10854	5	18.9	0.0
650	11746	11907	11901	11905	11915	11903	11906	6	19.0	0.0
700	12794	12978	12974	12976	12992	12976	12980	8	19.2	0.0
750	13853	14071	14070	14070	14090	14071	14075	10	19.4	0.0
800	14948	15197	15189	15195	15211	15199	15198	9	19.6	0.0

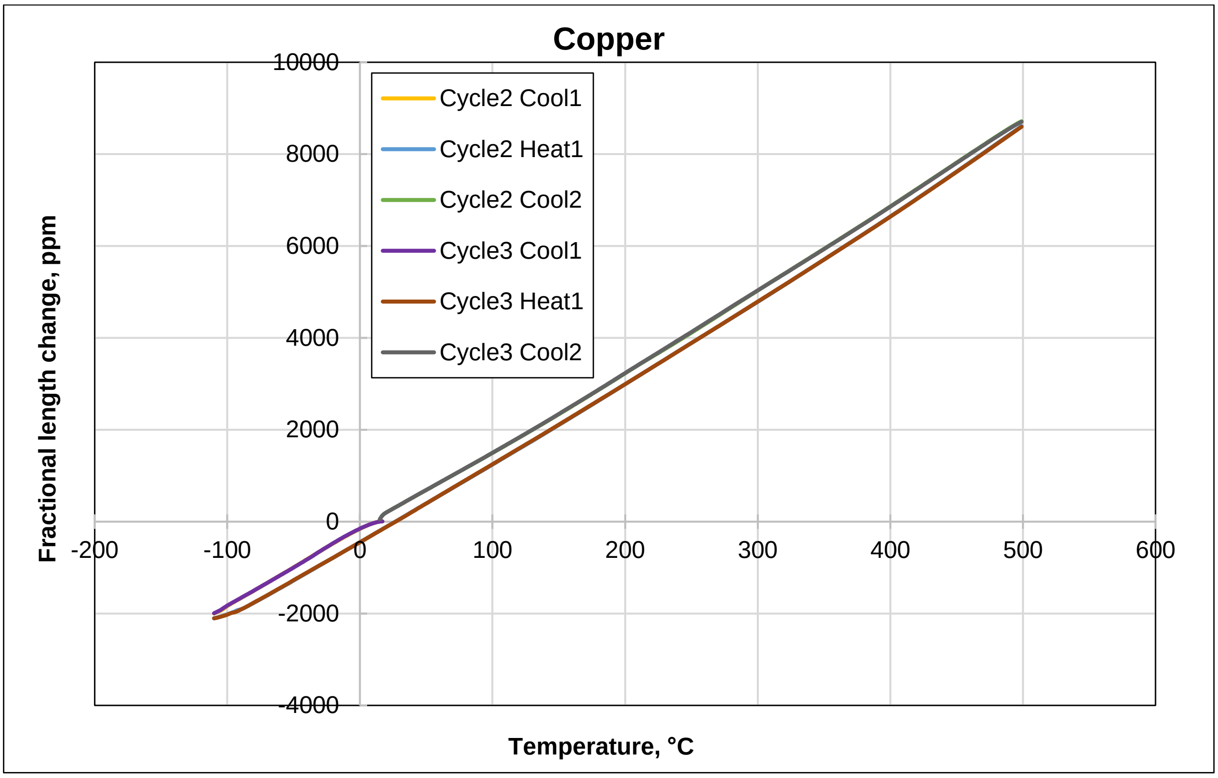


Figure 3: Linseis L75VS low-temperature dilatometer: Fractional length change results as a function of temperature for a copper sample over two heating and cooling cycles

Table 3: Linseis L75VS low-temperature dilatometer: Fractional length change and mean linear expansion coefficient from 25 °C as a function of temperature

Temp., °C	Fractional length change, ppm		Mean exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-1991	1	15.9	0.0
-50	-1250	8	16.7	-0.1
0	-421	3	16.8	-0.1
25	0	0	-	-
50	414	11	16.6	0.4
100	1243	33	16.6	0.4
150	2095	43	16.8	0.3
200	2983	39	17.0	0.2
250	3875	45	17.2	0.2
300	4781	36	17.4	0.1
350	5693	44	17.5	0.1
400	6622	51	17.7	0.1
450	7588	64	17.9	0.1
499	8531	113	18.0	0.2

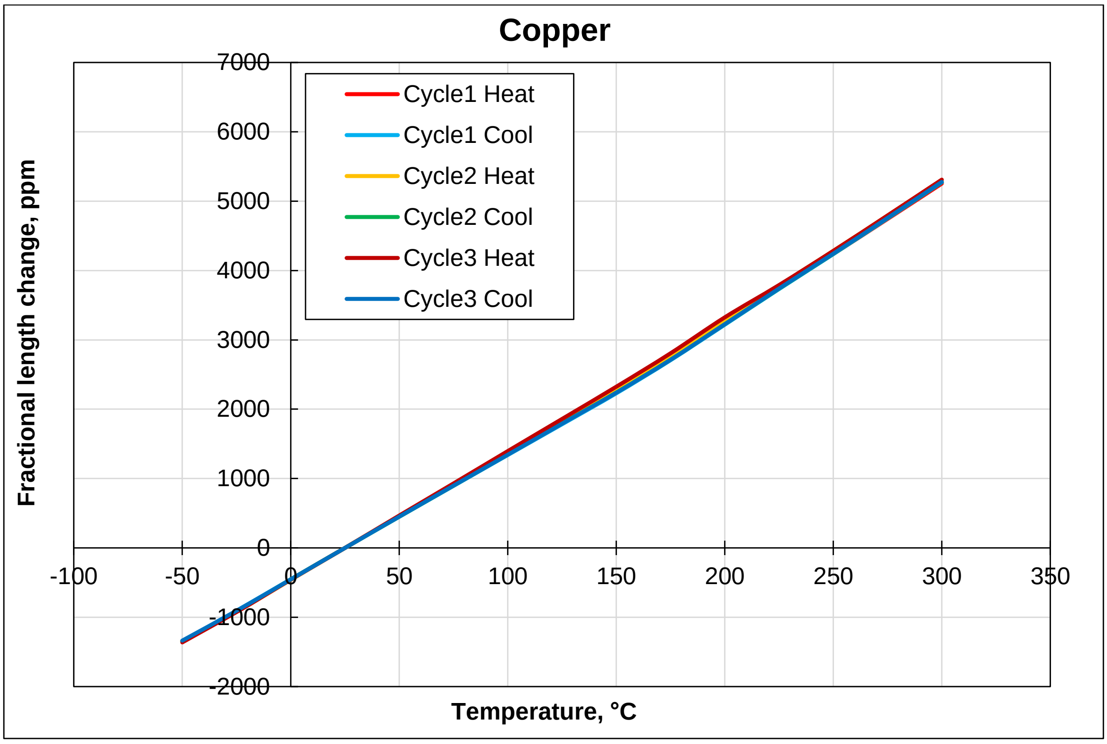


Figure 4: TMA: Fractional length change results as a function of temperature for a copper sample over three heating and cooling cycles.

Table 4: TMA: Fractional length change and mean linear expansion coefficient from 25 °C as a function of temperature

Temp., °C	Fractional length change from 25 °C, ppm								Expansion coeff. from 25 °C, 10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev.
-50	-1344	-1347	-1350	-1342	-1363	-1337	-1348	11	18.0	-0.1
-25	-924	-907	-922	-920	-925	-906	-918	8	18.4	-0.2
0	-459	-452	-461	-454	-458	-455	-457	3	18.3	-0.1
25	0	0	0	0	0	0	0	0	-	-
50	459	449	461	450	467	451	457	8	18.3	0.3
75	917	894	918	897	930	898	911	16	18.2	0.3
100	1378	1340	1374	1346	1396	1344	1365	25	18.2	0.3
125	1835	1783	1827	1794	1859	1791	1818	32	18.2	0.3
150	2294	2229	2281	2240	2324	2238	2271	41	18.2	0.3
175	2768	2706	2747	2713	2806	2712	2745	44	18.3	0.3
200	3276	3218	3260	3225	3324	3226	3259	47	18.6	0.3
225	3749	3733	3781	3738	3790	3741	3763	27	18.8	0.1
250	4238	4236	4283	4241	4283	4244	4263	24	18.9	0.1
275	4743	4752	4792	4756	4794	4758	4775	21	19.1	0.1
300	5253	5272	5306	5274	5310	5278	5292	19	19.2	0.1

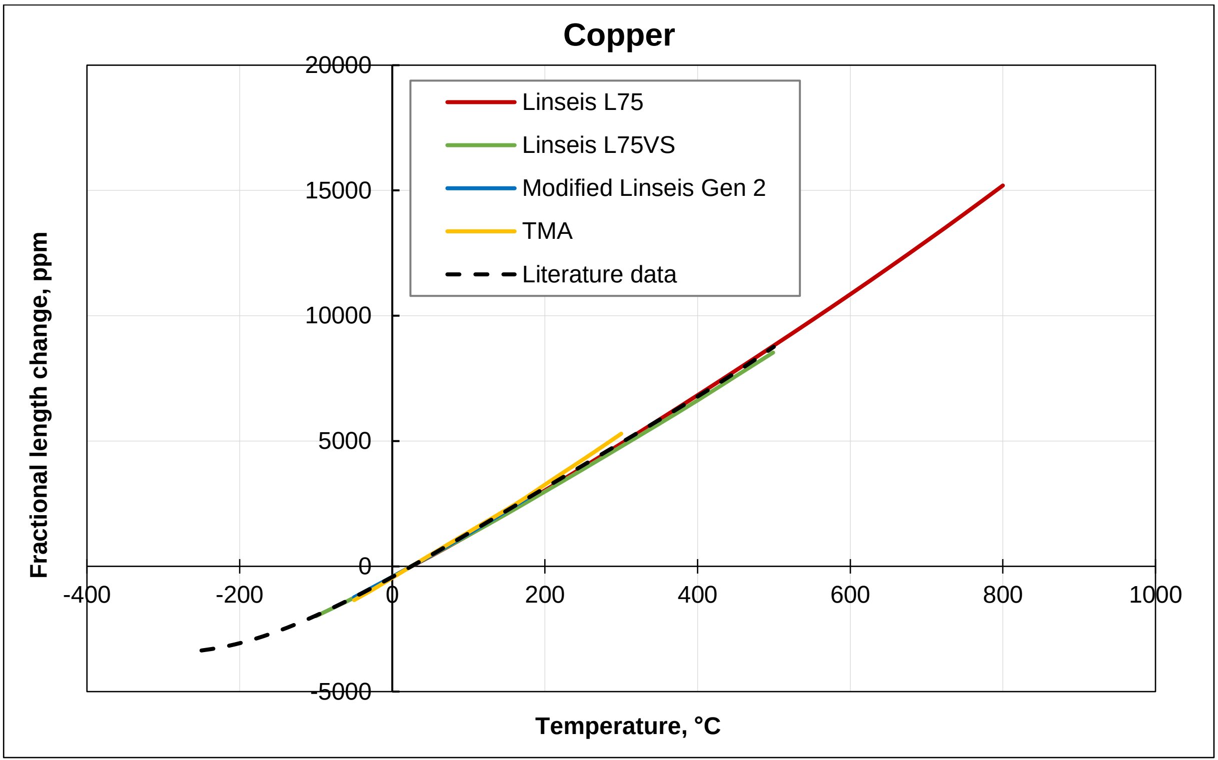


Figure 5: Fractional length change as a function of temperature comparing the four systems and including literature data[2].

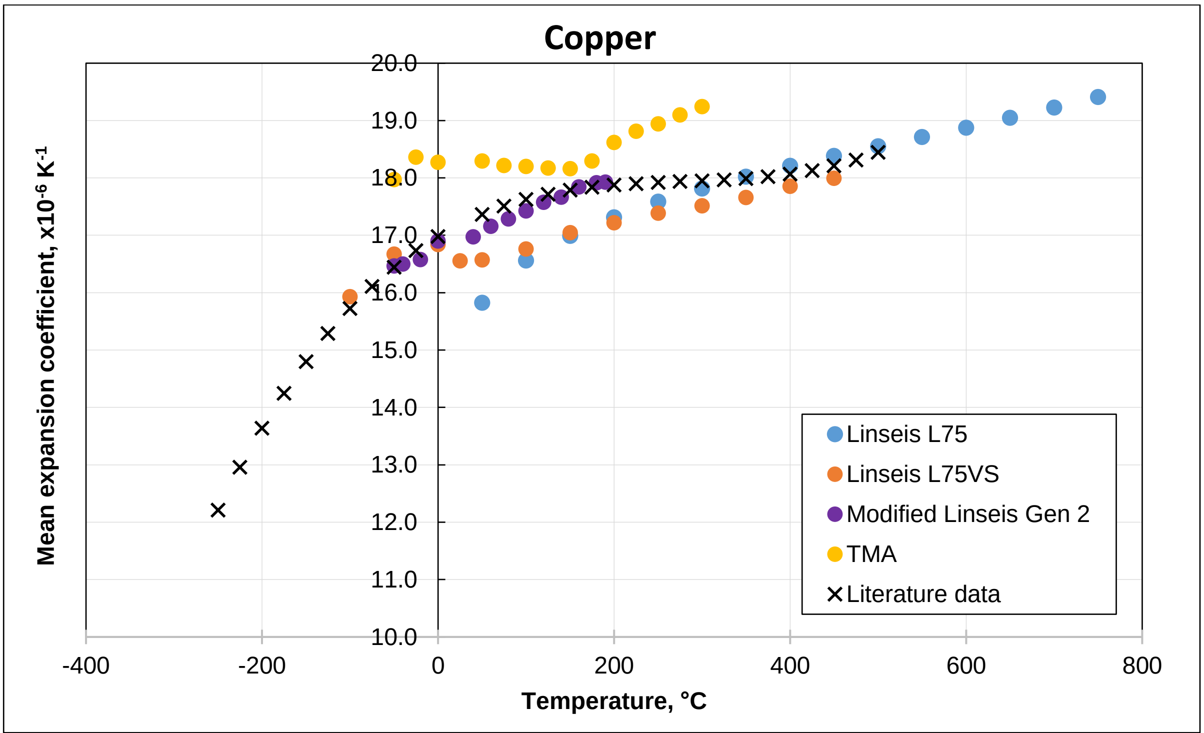


Figure 6: Mean linear expansion coefficient as a function of temperature for the four systems.

[2] Thermal expansion of copper from 20 to 800 K – Standard Reference Material 736, J. Appl. Phys., 41, 5096-5101 (1970)

Table 5: Fractional length change and mean linear expansion coefficient of copper as a function of temperature for the four systems

Linseis L75				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
25	0	0		
50	396	12	15.8	0.5
100	1242	14	16.6	0.2
150	2124	17	17.0	0.1
200	3030	11	17.3	0.1
250	3957	10	17.6	0.0
300	4900	7	17.8	0.0
350	5857	7	18.0	0.0
400	6829	6	18.2	0.0
450	7815	5	18.4	0.0
500	8813	4	18.6	0.0
550	9825	5	18.7	0.0
600	10854	5	18.9	0.0
650	11906	6	19.0	0.0
700	12980	8	19.2	0.0
750	14075	10	19.4	0.0
800	15198	9	19.6	0.0

Linseis L75VS				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-1991	1	15.9	0.0
-50	-1250	8	16.7	-0.1
0	-421	3	16.8	-0.1
25	0	0		
50	414	11	16.6	0.4
100	1243	33	16.6	0.4
150	2095	43	16.8	0.3
200	2983	39	17.0	0.2
250	3875	45	17.2	0.2
300	4781	36	17.4	0.1
350	5693	44	17.5	0.1
400	6622	51	17.7	0.1
450	7588	64	17.9	0.1
499	8531	113	18.0	0.2

Modified Linseis Gen 2				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-50	-1235	13	16.5	-0.2
-40	-1073	12	16.5	-0.2
-20	-746	10	16.6	-0.2
0	-423	31	16.9	-1.2
25	0	0	-	-
40	255	4	17.0	0.3
60	601	9	17.2	0.3
80	951	12	17.3	0.2
100	1307	14	17.4	0.2
120	1670	14	17.6	0.2
140	2032	29	17.7	0.2
160	2409	16	17.8	0.1
180	2777	13	17.9	0.1
190	2958	15	17.9	0.1

TMA				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-50	-1348	11	18.0	-0.1
-25	-918	8	18.4	-0.2
0	-457	3	18.3	-0.1
25	0	0	-	-
50	457	8	18.3	0.3
75	911	16	18.2	0.3
100	1365	25	18.2	0.3
125	1818	32	18.2	0.3
150	2271	41	18.2	0.3
175	2745	44	18.3	0.3
200	3259	47	18.6	0.3
225	3763	27	18.8	0.1
250	4263	24	18.9	0.1
275	4775	21	19.1	0.1
300	5292	19	19.2	0.1

3.2 PLATINUM

Platinum has also been widely used as a high-temperature reference material as it has a face-centred cubic crystal structure, has a high melting point, has no problems of oxidation, and does not have any phase changes between absolute zero and its melting point. However, the availability of sub-room-temperature data is limited. It has never been available as a certified reference material because of cost.

Fractional length change data are given in Figures 7 to 9 and Tables 6 to 8, with an intercomparison of averaged data given in Figure 10. Figure 11 shows a comparison of mean expansion coefficients. Table 10 gives an overall summary of data for the three systems in which it was measured.

Overall, there is a good agreement between the four instruments and with the reference data, as can be seen from Figure 10.

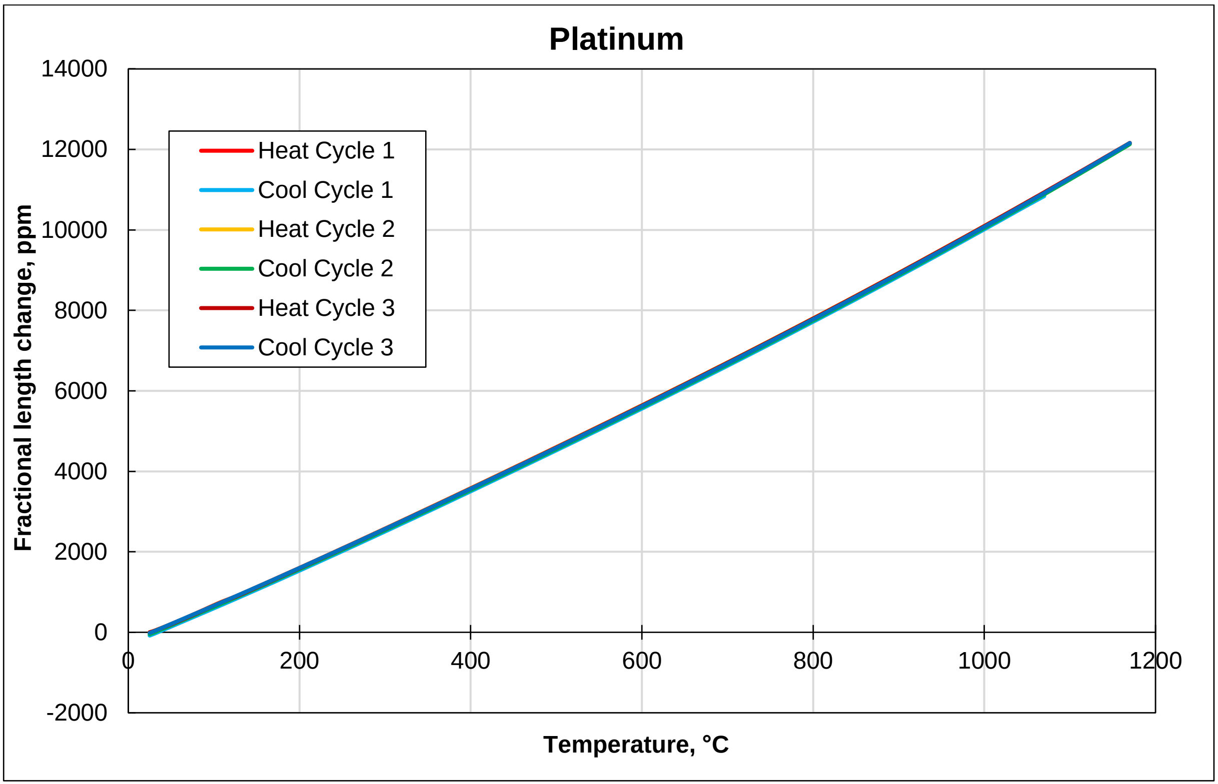


Figure 7: Linseis L75 high-temperature dilatometer: Fractional length change as a function of temperature for a copper sample over three heating and cooling cycles

Table 6: Linseis L75 high-temperature dilatometer: Fractional length change and mean expansion coefficient as a function of temperature

Temp., °C	Fractional length change from 25 °C								Exp. Coeff. from 25 °C, 10^{-6} K^{-1}	
	Cycle 1, ppm		Cycle 2, ppm		Cycle 3, ppm		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev
50	205	223	201	223	199	223	211	13	8.5	0.5
100	674	677	668	677	664	677	672	7	9.0	0.1
150	1123	1140	1118	1141	1115	1140	1129	14	9.0	0.1
200	1601	1614	1596	1615	1593	1614	1604	12	9.2	0.1
250	2086	2096	2081	2097	2078	2096	2088	10	9.3	0.0
300	2578	2586	2574	2586	2570	2586	2579	8	9.4	0.0
350	3076	3083	3072	3082	3068	3081	3076	7	9.5	0.0
400	3580	3584	3576	3583	3571	3582	3578	6	9.5	0.0
450	4088	4091	4085	4090	4080	4089	4086	5	9.6	0.0
500	4601	4602	4598	4600	4592	4599	4597	4	9.7	0.0
550	5118	5118	5116	5116	5110	5114	5114	3	9.7	0.0
600	5640	5640	5639	5638	5633	5636	5637	2	9.8	0.0
650	6167	6168	6169	6166	6163	6165	6166	3	9.9	0.0
700	6701	6704	6706	6701	6700	6700	6702	3	9.9	0.0
750	7240	7247	7250	7245	7244	7243	7246	3	10.0	0.0
800	7786	7798	7802	7797	7797	7795	7798	3	10.1	0.0
850	8338	8358	8362	8356	8357	8355	8357	3	10.1	0.0
900	8898	8927	8931	8926	8927	8924	8927	3	10.2	0.0
950	9469	9506	9511	9504	9507	9503	9506	3	10.3	0.0
1000	10047	10095	10097	10093	10095	10092	10094	2	10.4	0.0
1050	10632	10688	10692	10691	10693	10690	10691	2	10.4	0.0
1100	-	-	11292	11302	11301	11301	11299	4	10.5	0.0
1150	-	-	11896	11925	11916	11923	11915	13	10.6	0.0

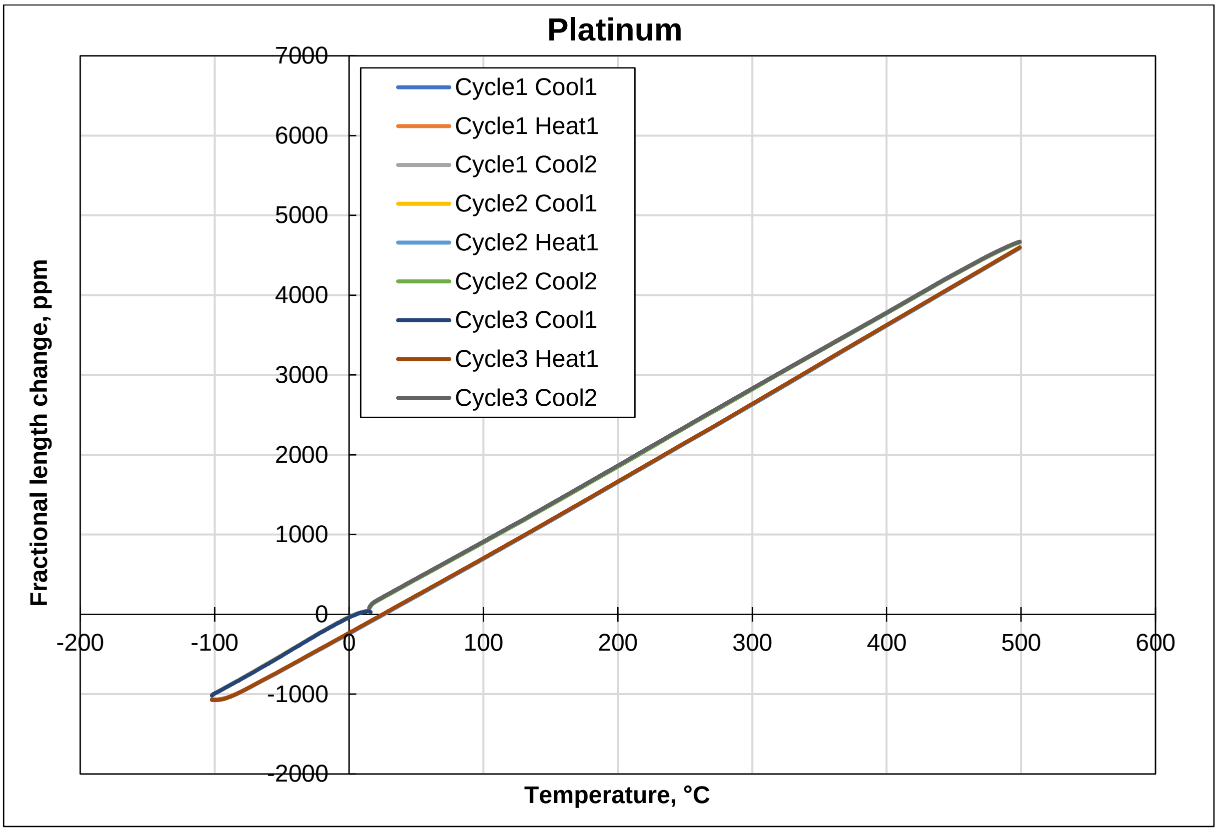


Figure 8: Linseis L75VS low-temperature dilatometer: Fractional length change as a function of temperature for a platinum sample over three heating and cooling cycles

Table 7: Linseis L75VS low-temperature dilatometer: Fractional length change and mean expansion coefficient as a function of temperature

Temp., °C	Fractional length change, ppm		Expansion coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-1065	9	8.5	-0.1
-50	-694	3	9.3	0.0
0	-232	2	9.3	-0.1
25	0	0	-	-
50	233	2	9.3	0.1
100	699	5	9.3	0.1
150	1173	9	9.4	0.1
200	1656	10	9.5	0.1
250	2142	11	9.5	0.0
300	2626	12	9.5	0.0
350	3112	22	9.6	0.1
400	3597	33	9.6	0.1
450	4082	39	9.6	0.1
499	4528	80	9.6	0.2

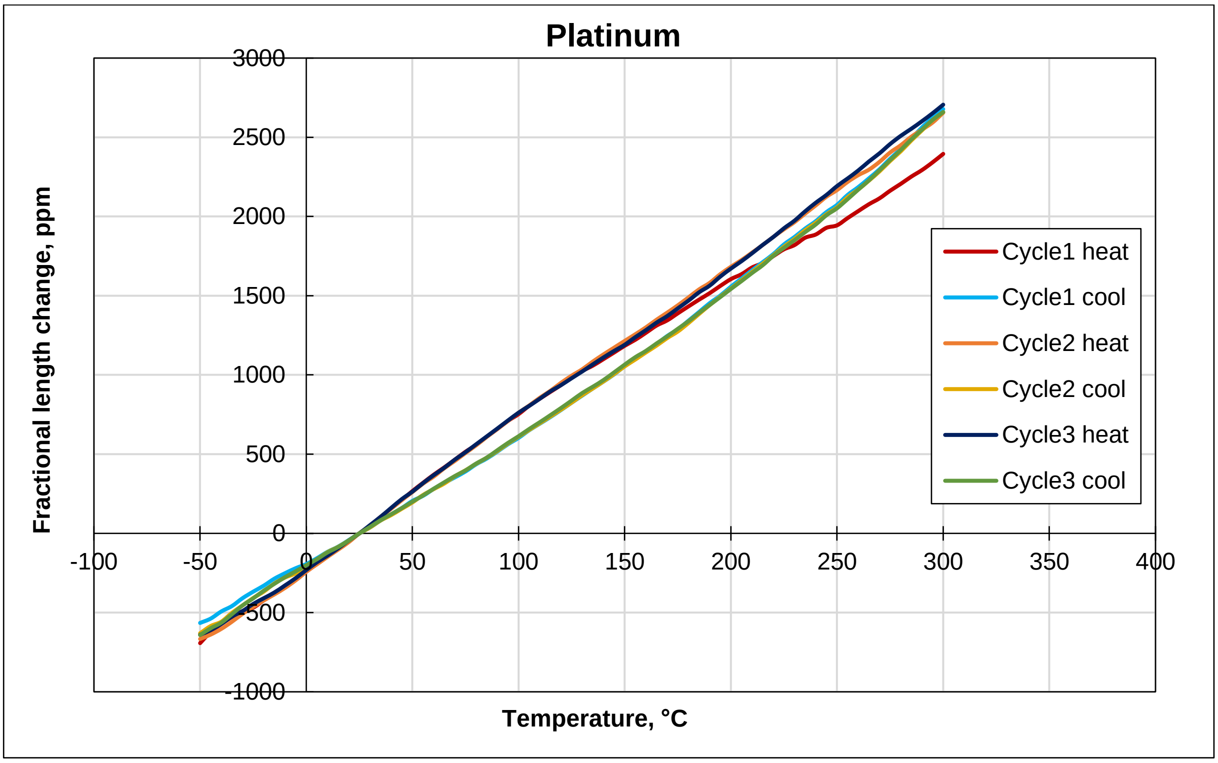


Figure 9: TMA: Fractional length change as a function of temperature for a copper sample over three heating and cooling cycles.

Table 8: TMA: Fractional length change and mean expansion coefficient as a function of temperature

Temp., °C	Fractional length change from 25 °C, ppm								Expansion coeff. from 25 °C, 10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev.
-50	-693	-565	-666	-630	-640	-642	-645	15	8.6	-0.2
-25	-469	-368	-464	-408	-446	-410	-432	28	8.6	-0.6
0	-242	-194	-242	-207	-232	-201	-220	19	8.8	-0.8
25	0	0	0	0	0	0	0	0	-	-
50	267	205	261	195	264	200	230	37	9.2	1.5
75	515	391	507	398	512	397	454	65	9.1	1.3
100	753	603	762	610	762	613	687	87	9.2	1.2
125	982	825	995	825	979	838	910	90	9.1	0.9
150	1184	1055	1215	1054	1190	1065	1131	83	9.0	0.7
175	1389	1288	1439	1274	1420	1291	1356	86	9.0	0.6
200	1605	1557	1683	1545	1671	1543	1610	77	9.2	0.4
225	1793	1824	1919	1810	1926	1802	1864	67	9.3	0.3
250	1944	2074	2167	2058	2192	2052	2117	73	9.4	0.3
275	2163	2364	2404	2350	2457	2356	2392	50	9.6	0.2
300	2395	2678	2655	2659	2706	2659	2670	24	9.7	0.1

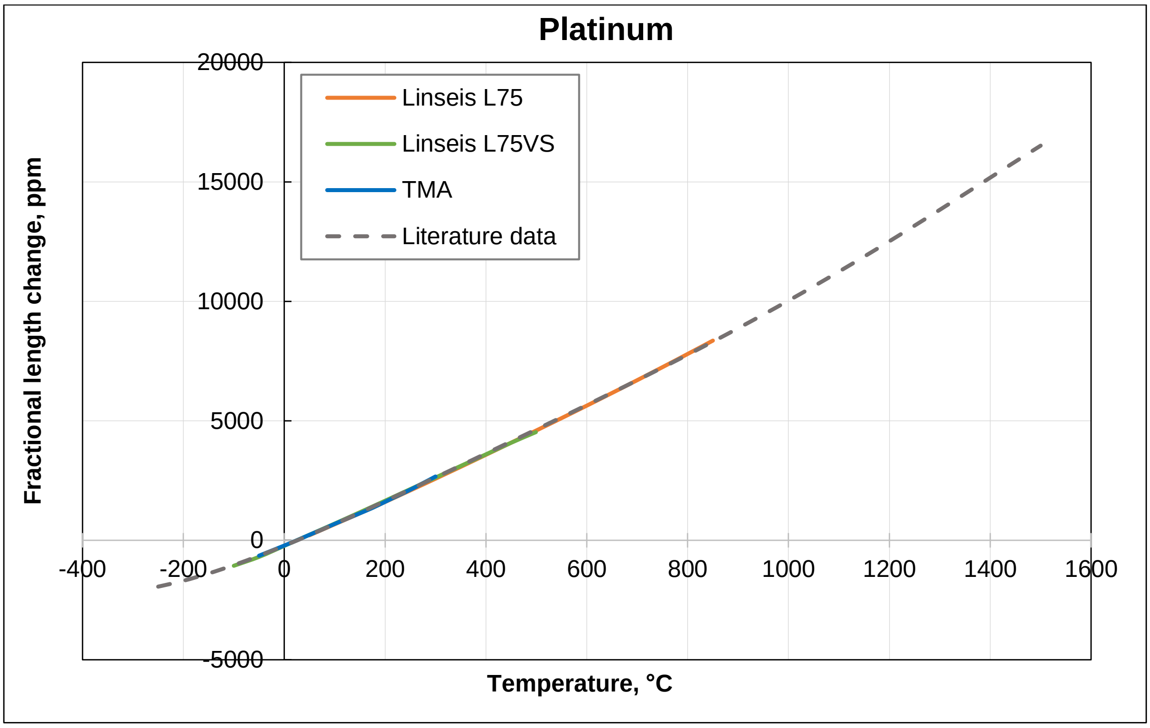


Figure 10: Comparison of the mean fractional length change as a function of temperature for a platinum sample using the three systems and including literature data[3].

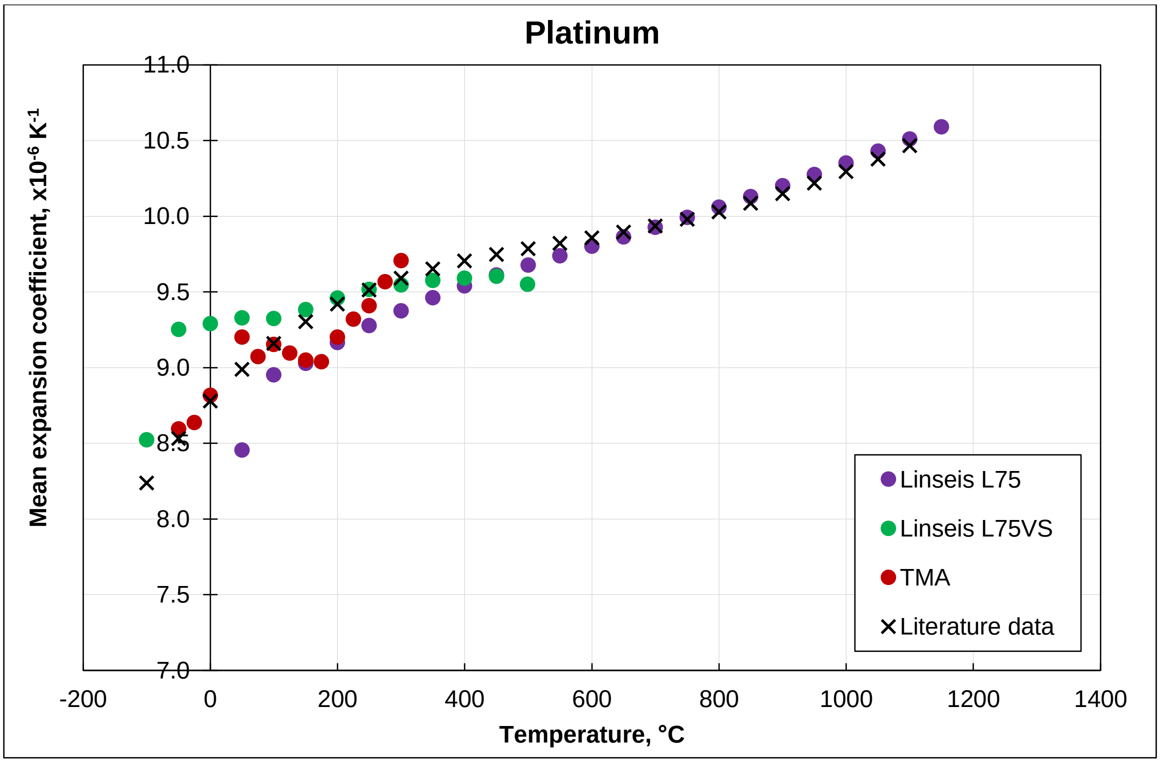


Figure 11: Comparison of the mean expansion coefficient as a function of temperature for a platinum sample using the three systems and including literature data.

[3] Kirby, R.K. Platinum—A thermal expansion reference material. *Int J Thermophys* **12**, 679–685 (1991).

Table 9: Fractional length change and mean linear expansion coefficient of platinum as a function of temperature for the three systems.

Linseis L75				
Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev
25	0	0		
50	211	13	8.5	0.5
100	672	7	9.0	0.1
150	1129	14	9.0	0.1
200	1604	12	9.2	0.1
250	2088	10	9.3	0.0
300	2579	8	9.4	0.0
350	3076	7	9.5	0.0
400	3578	6	9.5	0.0
450	4086	5	9.6	0.0
500	4597	4	9.7	0.0
550	5114	3	9.7	0.0
600	5637	2	9.8	0.0
650	6166	3	9.9	0.0
700	6702	3	9.9	0.0
750	7246	3	10.0	0.0
800	7798	3	10.1	0.0
850	8357	3	10.1	0.0
900	8927	3	10.2	0.0
950	9506	3	10.3	0.0
1000	10094	2	10.4	0.0
1050	10691	2	10.4	0.0
1100	11299	4	10.5	0.0
1150	11915	13	10.6	0.0

Linseis L75VS				
Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev
-100	-1065	8.7	8.5	-0.1
-50	-694	2.7	9.3	0.0
0	-232	1.6	9.3	-0.1
25	0	0.0		
50	233	1.5	9.3	0.1
100	699	5.0	9.3	0.1
150	1173	8.9	9.4	0.1
200	1656	10.2	9.5	0.1
250	2142	10.9	9.5	0.0
300	2626	12.4	9.5	0.0
350	3112	22.2	9.6	0.1
400	3597	32.7	9.6	0.1
450	4082	39.5	9.6	0.1
499	4528	79.9	9.6	0.2

TMA				
Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev
-50	-645	15	8.6	-0.2
-25	-432	28	8.6	-0.6
0	-220	19	8.8	-0.8
25	0	0		
50	230	37	9.2	1.5
75	454	65	9.1	1.3
100	687	87	9.2	1.2
125	910	90	9.1	0.9
150	1131	83	9.0	0.7
175	1356	86	9.0	0.6
200	1610	77	9.2	0.4
225	1864	67	9.3	0.3
250	2117	73	9.4	0.3
275	2392	50	9.6	0.2
300	2670	24	9.7	0.1

3.3 INCONEL 718

Inconel 718 (IN718) is a high-strength, corrosion resistant nickel chromium material used for components for liquid-fuelled rockets, parts for aircraft and land-based turbine engines and cryogenic tankage. It was selected as one of the candidate materials for this work as NPL routinely carries out measurements on nickel superalloys. However, it is not suitable as a true traceable reference material because of on-going phase changes at elevated temperatures.

The test-piece used in the present work comes from an annealed rod purchased from Goodfellow.

The results for the individual instruments are given in Figures 12 to 14 and are tabulated in Tables 10 to 12. As can be seen from the fractional length change plot in Figure 14, the new Linseis L75VS dilatometer produces a slight thermal hysteresis, and this also occurs in the set of results for the old Linseis system. The result is repeatable over two heating and cooling cycles.

Overall, there is a good agreement between the three instruments used to measure this material and the literature data, as can be seen from Figure 15.

In Figure 16 a comparison is made between the mean expansion coefficients determined from a 25 °C origin.

Table 13 gives an overall summary of the averaged data for each instrument.

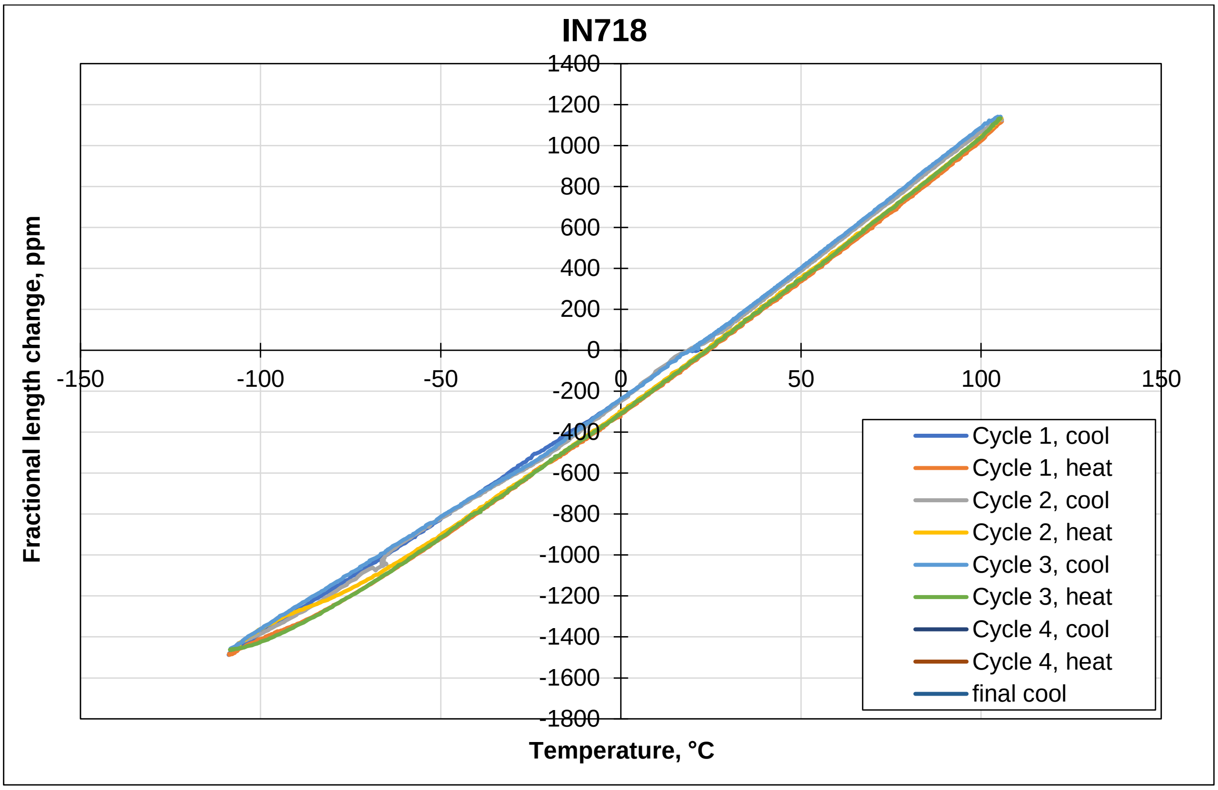


Figure 12: Modified Linseis Gen 2: Fractional length change as a function of temperature for a platinum sample over four heating and cooling cycles

Table 10: Modified Linseis Gen 2: Fractional length change and mean expansion coefficient as a function of temperature of IN718

Temp, °C	Fractional length change, ppm, from 20 °C											Mean exp. coeff. from 20 °C, x10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Cycle 4		Final cool	Mean from 20 °C	Std.dev.		
	Cool	Heat	Cool	Heat	Cool	Heat	Cool	Heat				Mean	Std.dev.
-100	-1386	-1353	-1396	-1318	-1365	-1375	-1417	-1348	-	-1374	35	11.5	-0.3
-80	-1168	-1192	-1198	-1163	-1147	-1201	-1237	-1191	-	-1189	32	11.9	-0.3
-60	-945	-979	-943	-970	-927	-985	-1002	-981	-	-965	28	12.1	-0.4
-40	-708	-742	-725	-737	-710	-743	-772	-741	-	-737	21	12.3	-0.3
-20	-472	-492	-521	-503	-500	-497	-514	-498	-	-507	10	12.7	-0.2
0	-247	-258	-260	-256	-242	-259	-257	-259	-	-255	7	12.7	-0.3
20	0	0	0	0	0	0	0	0	0	0	0	-	-
40	-	265	243	266	263	267	253	265	255	258	10	12.9	0.5
60	-	528	515	533	535	531	526	531	530	528	7	13.2	0.2
80	-	761	785	804	810	808	804	807	806	802	9	13.4	0.2
100	-	1080	1057	1087	1084	1089	1073	1089	1076	1078	13	13.5	0.2

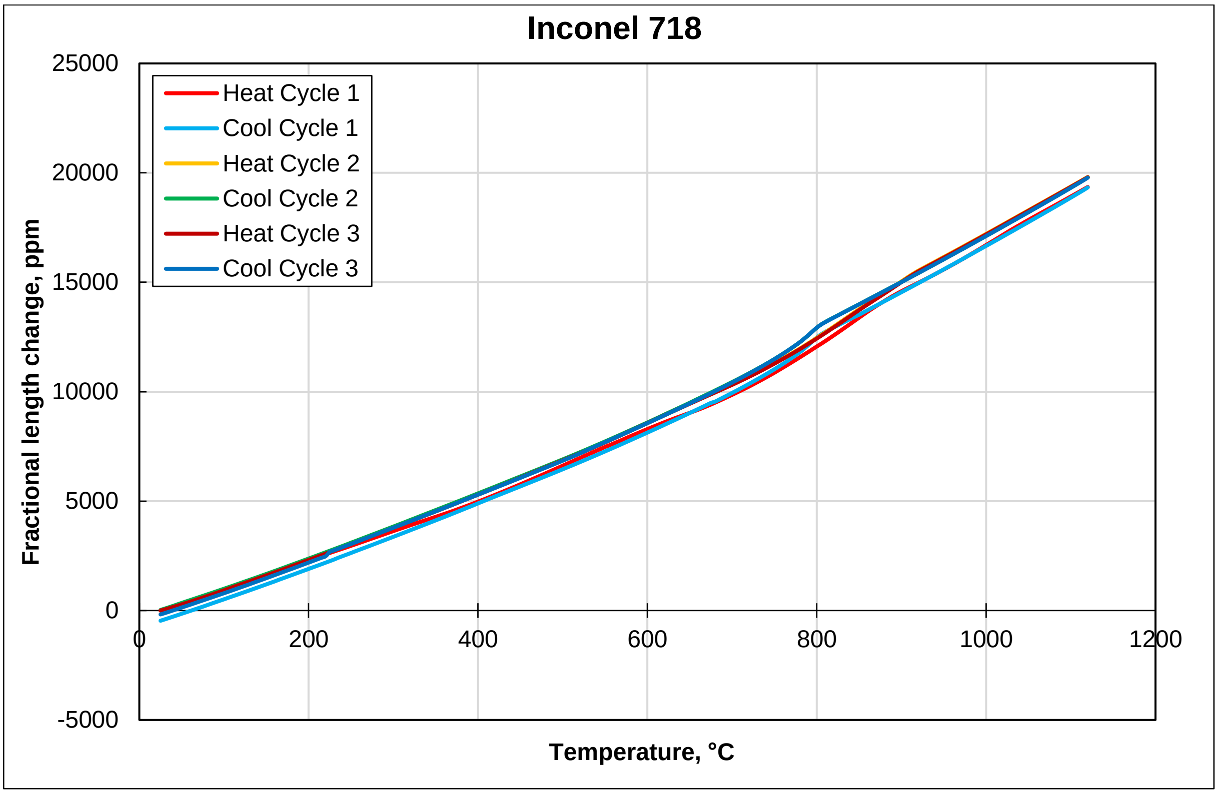


Figure 13: Linseis L75 high-temperature dilatometer: Fractional length change as a function of temperature for an IN718 sample over three heating and cooling cycles.

Table 11: Linseis L75 high-temperature dilatometer: Fractional length change and mean expansion coefficient as a function of temperature of IN718

Temp., °C	Fractional length change from 25 °C, ppm								Expansion coeff. from 25 °C, 10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev.
50	308	319	280	321	279	315	299	22	12.0	0.9
100	965	979	925	970	924	970	947	26	12.6	0.4
150	1629	1662	1606	1646	1605	1658	1629	27	13.0	0.2
200	2308	2370	2314	2349	2311	2370	2336	29	13.4	0.2
250	2960	3098	3041	3073	3036	3220	3092	87	13.7	0.4
300	3632	3835	3782	3811	3776	3961	3833	87	13.9	0.3
350	4281	4587	4538	4565	4531	4712	4586	85	14.1	0.3
400	4977	5364	5307	5328	5296	5473	5351	82	14.3	0.2
450	5769	6140	6086	6101	6075	6242	6126	78	14.4	0.2
500	6619	6922	6871	6881	6862	7025	6910	77	14.5	0.2
550	7474	7739	7696	7701	7682	7871	7738	89	14.7	0.2
600	8292	8598	8582	8566	8566	8740	8614	84	15.0	0.1
650	9033	9493	9469	9468	9449	9643	9507	91	15.2	0.1
700	9858	10410	10334	10415	10314	10595	10414	128	15.4	0.2
750	10869	11492	11305	11467	11283	11667	11431	178	15.8	0.2
800	12065	12943	12459	12898	12430	13103	12723	332	16.4	0.4
850	13383	13987	13769	13971	13730	14168	13910	202	16.9	0.2
900	14590	15012	15041	14990	15008	15185	15056	89	17.2	0.1
950	15585	16056	16156	16028	16125	16227	16134	83	17.4	0.1
1000	16702	17129	17210	17094	17186	17293	17196	82	17.6	0.1
1050	17849	18218	18289	18184	18268	18380	18280	81	17.8	0.1
1100	18919	19334	19373	19299	19358	19494	19381	82	18.0	0.1

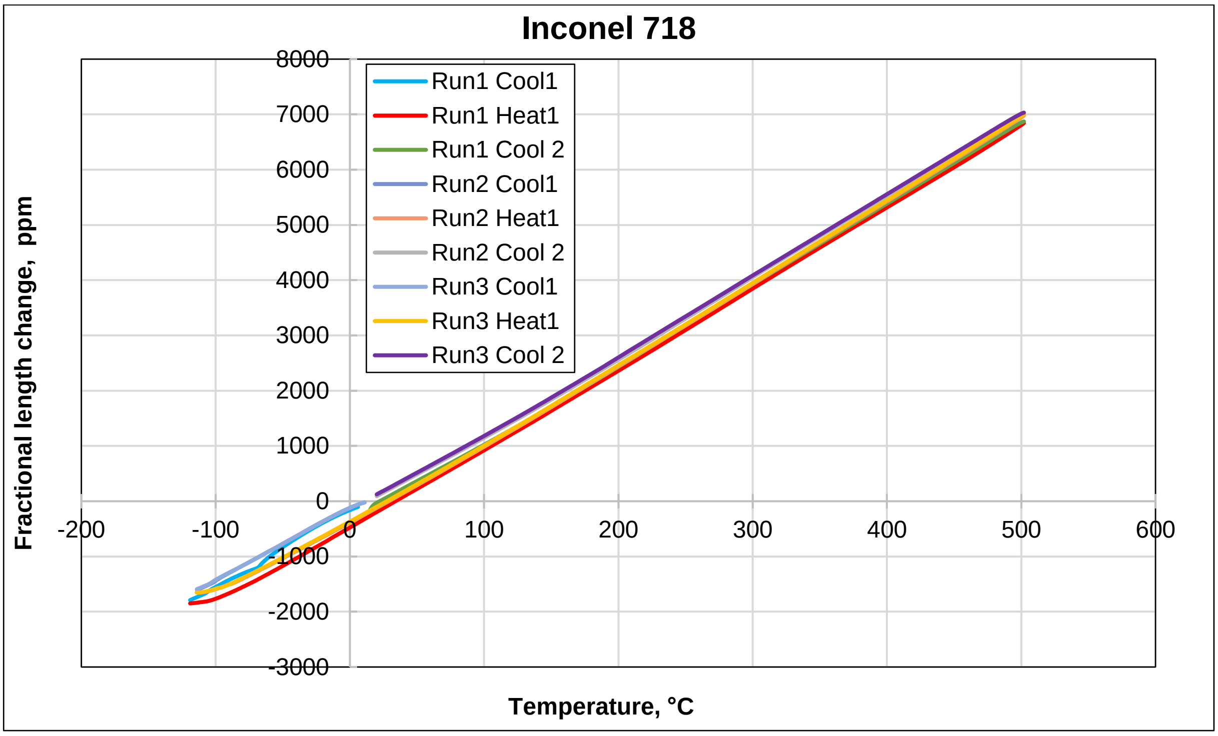


Figure 14: Linseis L75VS low-temperature dilatometer: Fractional length change as a function of temperature for an IN718 sample over three heating and cooling cycles.

Table 12: Linseis L75VS low-temperature dilatometer: Fractional length change and mean expansion coefficient as a function of temperature of IN718

Temp., °C	Fractional length change, ppm		Mean exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-1545	-1	12.4	0.0
-50	-983	-1	13.1	0.0
0	-334	0	13.4	0.0
25	0	0		
50	335	0	13.4	0.0
100	1016	1	13.5	0.0
150	1720	1	13.8	0.0
200	2451	1	14.0	0.0
250	3193	2	14.2	0.0
300	3940	2	14.3	0.0
350	4685	2	14.4	0.0
400	5424	3	14.5	0.0
450	6167	3	14.5	0.0
500	6912	3	14.6	0.0

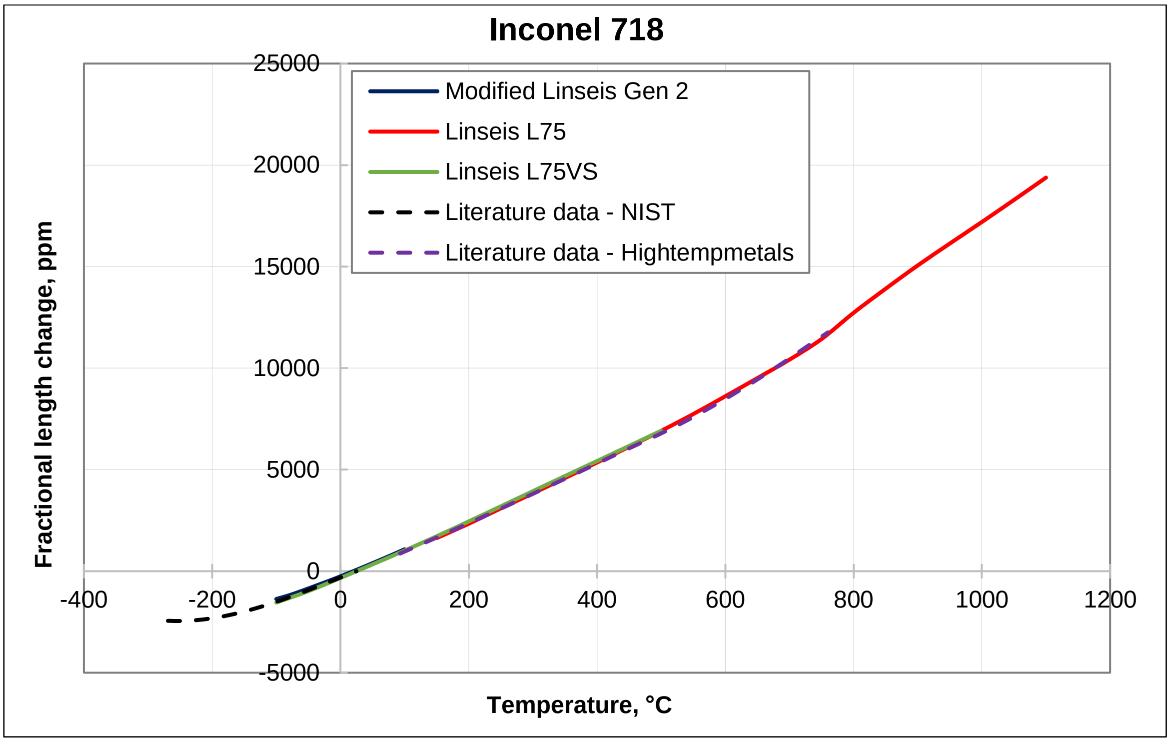


Figure 15: Comparison of the mean fractional length change as a function of temperature for a IN718 sample using the three systems and including literature data [4], [5].

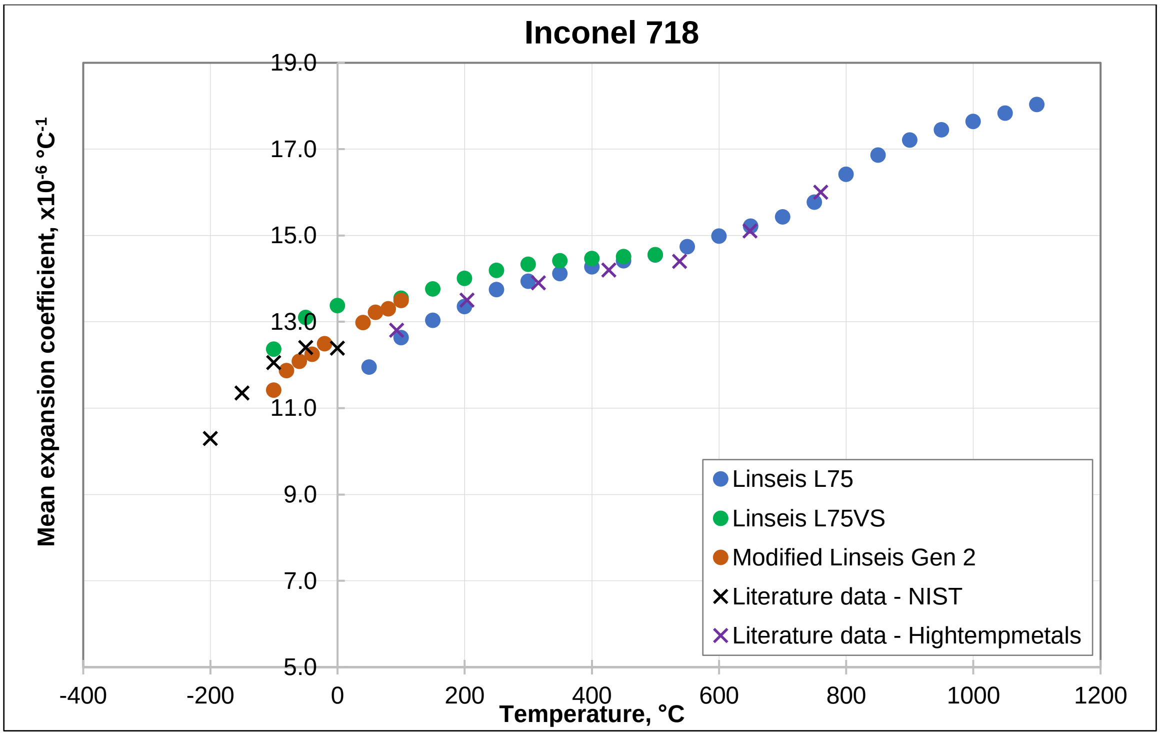


Figure 16: Comparison of the mean expansion coefficient as a function of temperature for a platinum sample using the three systems and including literature data.

[4] https://trc.nist.gov/cryogenics/materials/Iconel%20718/Inconel718_rev.htm
[5] https://www.hightempmetals.com/techdata/hitempInconel718data.php

Table 13: Fractional length change and mean linear expansion coefficient of IN718 as a function of temperature for the three systems

Linseis L75				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
25	0	0		
50	299	22	12.0	0.9
100	947	26	12.6	0.4
150	1629	27	13.0	0.2
200	2336	29	13.4	0.2
250	3092	87	13.7	0.4
300	3833	87	13.9	0.3
350	4586	85	14.1	0.3
400	5351	82	14.3	0.2
450	6126	78	14.4	0.2
500	6910	77	14.5	0.2
550	7738	89	14.7	0.2
600	8614	84	15.0	0.1
650	9507	91	15.2	0.1
700	10414	128	15.4	0.2
750	11431	178	15.8	0.2
800	12723	332	16.4	0.4
850	13910	202	16.9	0.2
900	15056	89	17.2	0.1
950	16134	83	17.4	0.1
1000	17196	82	17.6	0.1
1050	18280	81	17.8	0.1
1100	19381	82	18.0	0.1

Linseis L75VS				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-1545	-1	12.4	0.0
-50	-983	-1	13.1	0.0
0	-334	0	13.4	0.0
25	0	0		
50	335	0	13.4	0.0
100	1016	1	13.5	0.0
150	1720	1	13.8	0.0
200	2451	1	14.0	0.0
250	3193	2	14.2	0.0
300	3940	2	14.3	0.0
350	4685	2	14.4	0.0
400	5424	3	14.5	0.0
450	6167	3	14.5	0.0
500	6912	3	14.6	0.0

Modified Linseis Gen 2				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-1370	31	11.4	-0.3
-80	-1187	28	11.9	-0.3
-60	-967	26	12.1	-0.3
-40	-735	21	12.2	-0.3
-20	-500	15	12.5	-0.4
0	-255	7	12.7	-0.3
20	0	0		
40	260	9	13.0	0.4
60	529	6	13.2	0.2
80	798	17	13.3	0.3
100	1079	11	13.5	0.1

3.4 STAINLESS STEEL 316L

Stainless steel 316L is the second most commonly used austenitic steel after 304 stainless steel.

The test-piece used in the present work comes from Smiths Metals and it is AMINOX AS1 (1.4404 ASTM A479 316L) stainless steel.

The results for the individual instruments are given in Figures 17 to 19 and are tabulated in Tables 14 to 16. As can be seen from the fractional length change comparison plot in Figure 20, the new Linseis L75VS dilatometer produces higher fractional length change values above room temperature and lower below room temperature. The coefficient of thermal expansion is also higher than the literature values. As for previous experiments, the dilatometer produces a slight thermal hysteresis. The result is repeatable over two heating and cooling cycles. There is a good agreement between the high temperature Linseis dilatometer and the literature values.

In Figure 21 a comparison is made between the mean expansion coefficients determined from a 25 °C origin.

Table 17 gives an overall summary of the averaged data for each instrument.

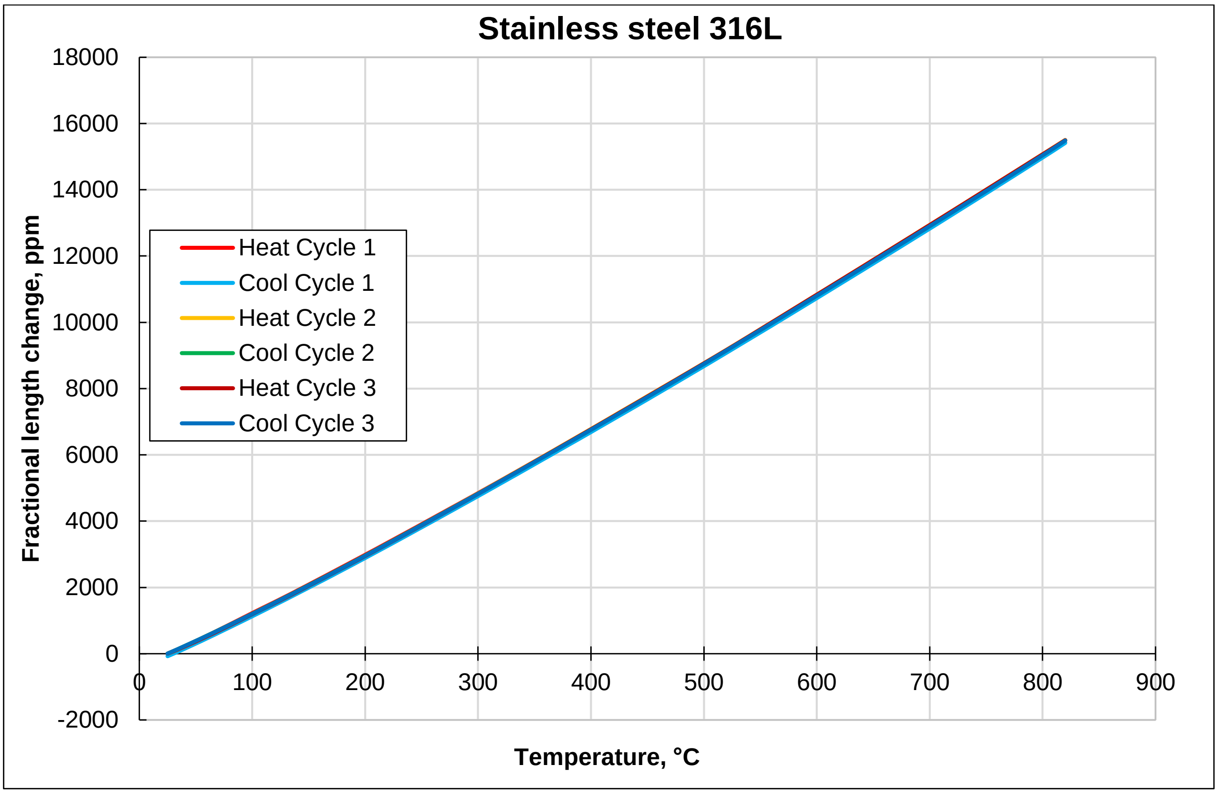


Figure 17: Linseis L75 high-temperature dilatometer: Fractional length change results for a stainless steel 316L sample using the high temperature Linseis L75 dilatometer over three heating and cooling cycles.

Table 14: Linseis L75 high-temperature dilatometer: Fractional length change and mean linear expansion coefficient from Linseis L75 dilatometer.

Temp., °C	Fractional length change from 25 °C, ppm								Expansion coeff from 25 °C, 10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev
50	382	381	371	381	368	382	375	7	15.0	0.3
100	1224	1205	1201	1197	1192	1196	1197	4	16.0	0.0
150	2083	2069	2057	2061	2048	2056	2056	5	16.4	0.0
200	2983	2963	2956	2955	2947	2951	2952	4	16.9	0.0
250	3906	3890	3883	3881	3874	3877	3879	4	17.2	0.0
300	4844	4832	4834	4823	4824	4821	4825	6	17.5	0.0
350	5801	5793	5801	5782	5792	5781	5789	9	17.8	0.0
400	6771	6764	6780	6754	6771	6756	6765	12	18.0	0.0
450	7749	7750	7771	7740	7760	7742	7754	15	18.2	0.0
500	8728	8752	8765	8742	8760	8743	8752	12	18.4	0.0
550	9727	9772	9790	9763	9786	9764	9776	14	18.6	0.0
600	10764	10806	10833	10799	10832	10800	10816	19	18.8	0.0
650	11811	11850	11882	11844	11880	11846	11863	21	19.0	0.0
700	12866	12903	12937	12898	12935	12900	12917	22	19.1	0.0
750	13932	13969	14001	13961	13999	13962	13981	22	19.3	0.0
800	14997	15048	15071	15039	15068	15040	15055	18	19.4	0.0

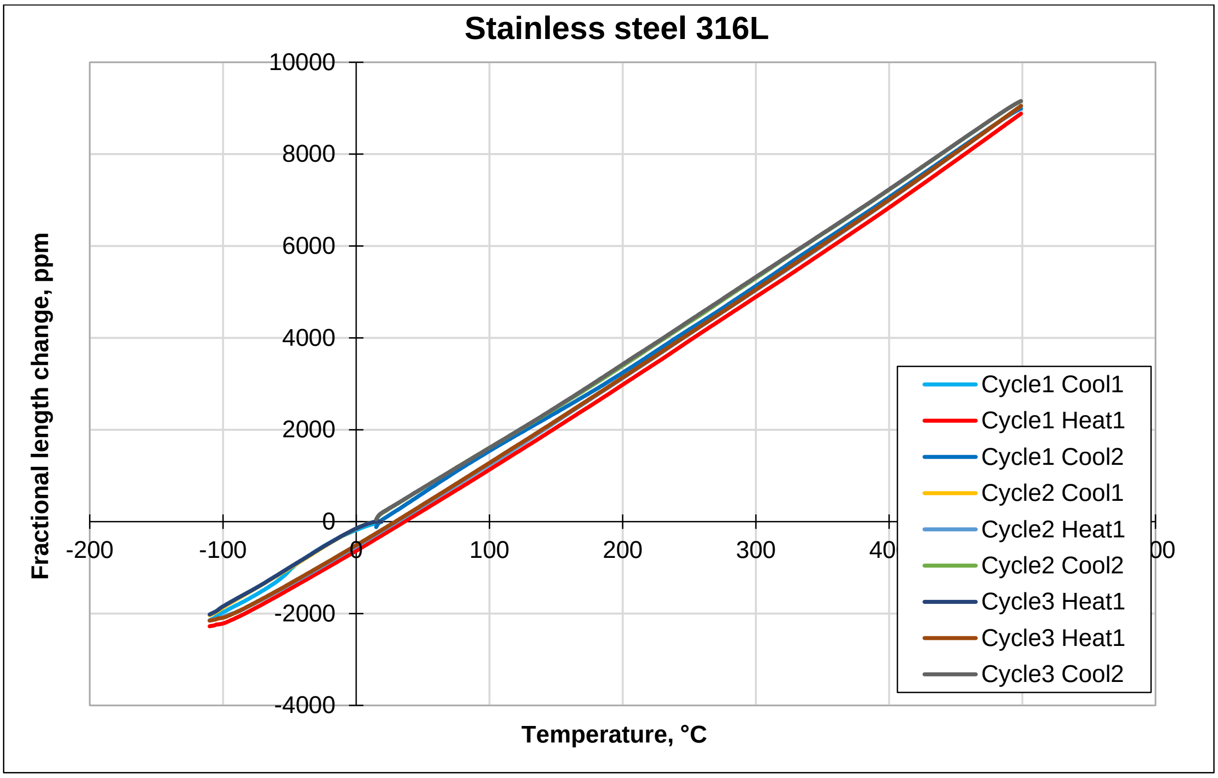


Figure 18: Linseis L75VS low-temperature dilatometer: Fractional length change results for a stainless steel 316L sample using the high temperature Linseis L75VS dilatometer over three heating and cooling cycles.

Table 15: Linseis L75VS low-temperature dilatometer: Fractional length change and mean linear expansion coefficient from Linseis L75VS low temperature dilatometer.

Temp., °C	Fractional length change, ppm		Mean exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-2001	26	16.0	-0.2
-50	-1271	5	17.0	-0.1
0	-437	1	17.5	0.0
25	0	0	-	-
50	447	12	17.9	0.5
100	1343	30	17.9	0.4
150	2235	39	17.9	0.3
200	3159	56	18.1	0.3
250	4112	62	18.3	0.3
300	5066	65	18.4	0.2
350	6028	69	18.5	0.2
400	7002	81	18.7	0.2
450	8013	96	18.9	0.2
499	8989	146	19.0	0.3

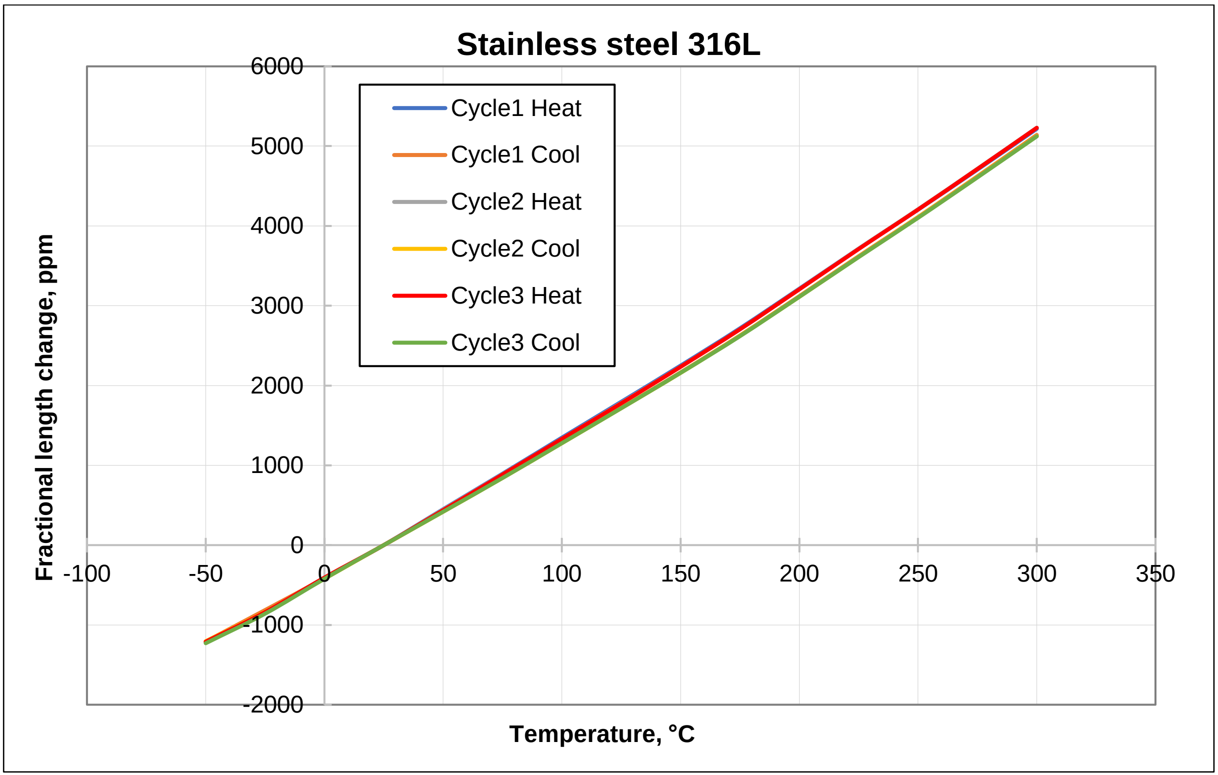


Figure 19: TMA: Fractional length change results for a stainless steel 316L sample using the TMA over three heating and cooling cycles.

Table 16: TMA: Fractional length change and mean linear expansion coefficient from the TMA for stainless steel 316L.

Temp., °C	Fractional length change from 25 °C, ppm								Expansion coeff. from 25 °C, 10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev.
-50	-1218	-1208	-1208	-1204	-1211	-1230	-1213	11	16.2	-0.1
-25	-856	-814	-843	-836	-840	-859	-844	10	16.9	-0.2
0	-413	-414	-407	-422	-408	-422	-415	8	16.6	-0.3
25	0	0	0	0	0	0	0	0	-	-
50	450	423	445	418	438	419	430	13	17.2	0.5
75	895	847	888	841	884	842	864	26	17.3	0.5
100	1347	1283	1332	1277	1331	1278	1304	31	17.4	0.4
125	1796	1721	1782	1716	1777	1716	1748	37	17.5	0.4
150	2249	2165	2236	2158	2235	2159	2197	44	17.6	0.4
175	2717	2627	2705	2622	2705	2619	2663	49	17.8	0.3
200	3213	3120	3207	3112	3206	3112	3159	55	18.1	0.3
225	3715	3620	3715	3612	3711	3610	3662	59	18.3	0.3
250	4204	4112	4209	4106	4205	4101	4155	60	18.5	0.3
275	4706	4622	4716	4612	4713	4608	4662	60	18.6	0.2
300	5217	5140	5231	5127	5229	5123	5177	61	18.8	0.2

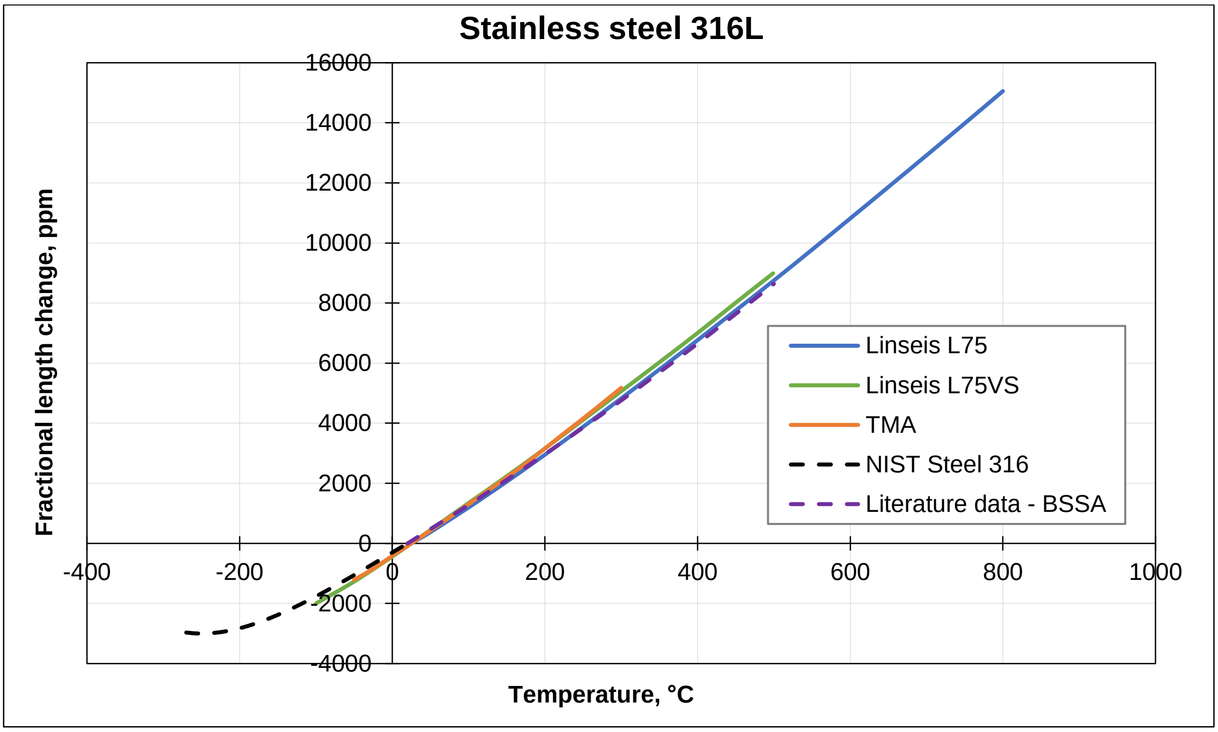


Figure 20: Comparison of the mean fractional length change as a function of temperature for a stainless steel 316L sample using the three systems and including literature data. [6], [7]

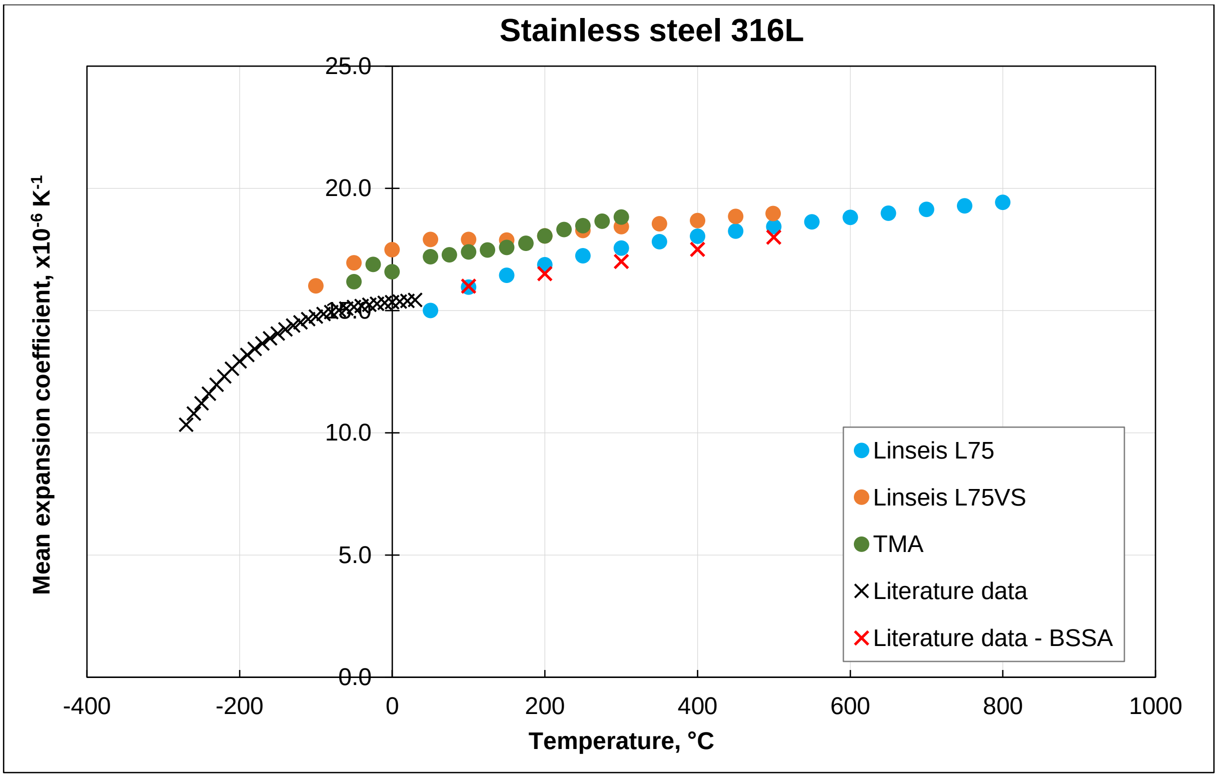


Figure 21: Comparison of the mean expansion coefficient as a function of temperature for a stainless steel 316 sample using the three systems and including literature data.

[6] https://trc.nist.gov/cryogenics/materials/316Stainless/316Stainless_rev.htm
[7] https://bssa.org.uk/bssa_articles/elevated-temperature-physical-properties-of-stainless-steels/

Table 17: Summary of averaged stainless steel 316L data for the three systems

Linseis L75				
Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
25	0	0	-	-
50	375	7	15.0	0.3
100	1197	4	16.0	0.0
150	2056	5	16.4	0.0
200	2952	4	16.9	0.0
250	3879	4	17.2	0.0
300	4825	6	17.5	0.0
350	5789	9	17.8	0.0
400	6765	12	18.0	0.0
450	7754	15	18.2	0.0
500	8752	12	18.4	0.0
550	9776	14	18.6	0.0
600	10816	19	18.8	0.0
650	11863	21	19.0	0.0
700	12917	22	19.1	0.0
750	13981	22	19.3	0.0
800	15055	18	19.4	0.0

Linseis L75VS				
Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-2001	26	16.0	-0.2
-50	-1271	5	17.0	-0.1
0	-437	1	17.5	0.0
25	0	0	-	-
50	447	12	17.9	0.5
100	1343	30	17.9	0.4
150	2235	39	17.9	0.3
200	3159	56	18.1	0.3
250	4112	62	18.3	0.3
300	5066	65	18.4	0.2
350	6028	69	18.5	0.2
400	7002	81	18.7	0.2
450	8013	96	18.9	0.2
499	8989	146	19.0	0.3

TMA				
Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-50	-1213	11	16.2	-0.1
-25	-844	10	16.9	-0.2
0	-415	8	16.6	-0.3
25	0	0	-	-
50	430	13	17.2	0.5
75	864	26	17.3	0.5
100	1304	31	17.4	0.4
125	1748	37	17.5	0.4
150	2197	44	17.6	0.4
175	2663	49	17.8	0.3
200	3159	55	18.1	0.3
225	3662	59	18.3	0.3
250	4155	60	18.5	0.3
275	4662	60	18.6	0.2
300	5177	61	18.8	0.2

3.5 PYROCERAM 9606

Pyroceram 9606 is a cordierite-based glass-ceramic which has been used to create a BCR reference material (BCR-724a) for thermal conductivity, associated with which are data for thermal expansion. However, there are no data below room temperature available in that certified work, only from the manufacturer, Corning Glass Works. This material can be subject to small variations from batch to batch, so is not ideal as a low-temperature reference material, but is representative of a low thermal conductivity, low thermal expansion material. The test-pieces used in the present work come from the BCR-certified batch.

In Figure 26 and 27 a comparison is made between the fractional length and the mean expansion coefficients determined from a 25 °C origin.

Table 22 gives an overall summary of the averaged data for each instrument.

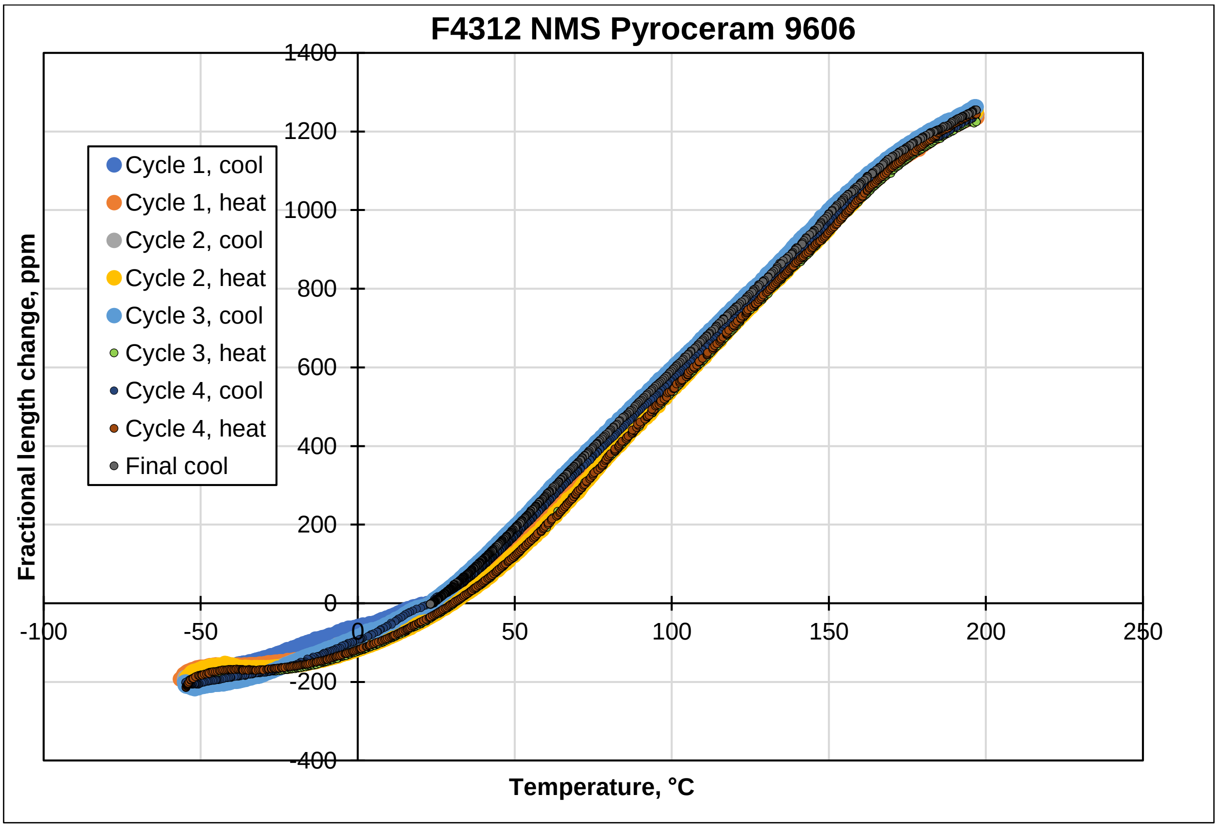


Figure 22: Modified Linseis Gen 2: Fractional length change as a function of temperature for a Pyroceram 9606 sample over four heating and cooling cycles

Table 18: Modified Linseis Gen 2: Fractional length change and mean expansion coefficient as a function of temperature of Pyroceram 9606

Temp, °C	Fractional length change, ppm, from 25 °C											Mean expansion coeff. from 25 °C, 10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Cycle 4		Final cool	Mean from 25 °C	Std.dev.		
	Cool	Heat	Cool	Heat	Cool	Heat	Cool	Heat				Mean	Std.dev.
-50	-178	-158	-207	-143	-223	-154	-218	-157	-	-180	32	2.4	-0.4
-40	-161	-152	-166	-133	-211	-147	-202	-142	-	-164	28	2.5	-0.4
-20	-115	-138	-152	-132	-159	-136	-171	-133	-	-142	18	3.2	-0.4
0	-63	-93	-99	-93	-96	-93	-108	-92	-	-92	13	3.7	-0.5
20	-6	-22	-21	-23	-21	-33	-25	-21	-30	-22	8	4.5	-1.5
25	0	0	0	0	0	0	0	0	0	0	0	-	-
40	-	79	101	81	104	81	82	80	104	89	12	5.9	0.8
60	-	227	270	226	268	226	239	134	267	232	44	6.6	1.3
80	-	398	428	401	429	400	399	401	428	411	15	7.5	0.3
100	-	563	581	570	586	565	554	568	584	571	11	7.6	0.2
120	-	730	734	736	743	731	710	735	741	733	10	7.7	0.1
140	-	892	892	899	905	893	870	896	897	893	10	7.8	0.1
160	-	1047	1044	1064	1063	1056	1035	1057	1059	1053	10	7.8	0.1
180	-	1173	1168	1200	1180	1187	1154	1192	1178	1179	14	7.6	0.1
190	-	1221	1210	1249	1221	1234	1196	1247	1220	1225	18	7.4	0.1

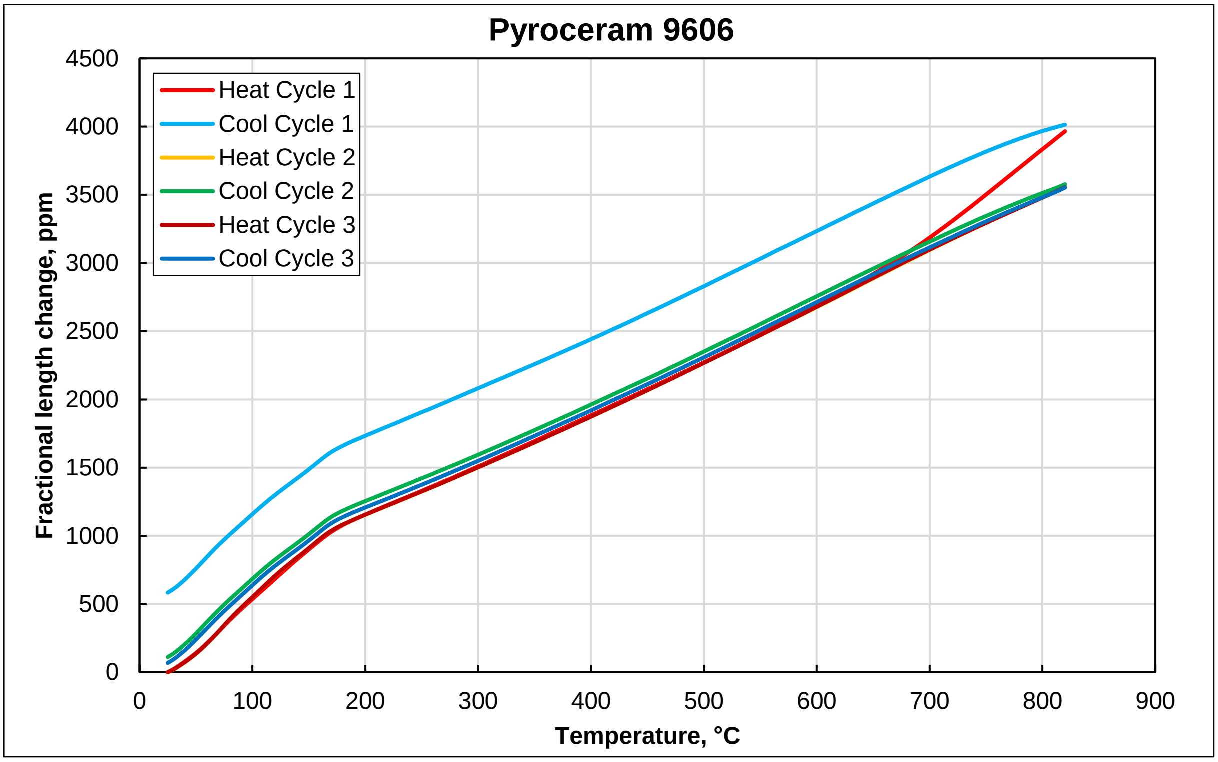


Figure 23: Linseis L75 high-temperature dilatometer: Fractional length change as a function of temperature for a Pyroceram 9606 sample over three heating and cooling cycles [8]

Table 19: Linseis L75 high-temperature dilatometer: Fractional length change and mean expansion coefficient as a function of temperature of Pyroceram 9606.

Temp., °C	Fractional length change from 25 °C, ppm								Expansion coeff. from 25 °C, 10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev.
50	141	176	135	173	136	171	154	21	6.1	0.8
100	536	575	550	573	551	570	561	12	7.5	0.2
150	900	903	909	901	910	897	904	6	7.2	0.0
200	1155	1150	1154	1143	1155	1140	1148	7	6.6	0.0
250	1329	1323	1324	1311	1326	1307	1317	9	5.9	0.0
300	1509	1498	1501	1483	1502	1481	1492	11	5.4	0.0
350	1694	1675	1684	1664	1685	1664	1674	12	5.2	0.0
400	1884	1858	1873	1851	1874	1851	1862	13	5.0	0.0
450	2075	2049	2068	2042	2068	2043	2055	14	4.8	0.0
500	2272	2245	2268	2239	2267	2240	2254	16	4.7	0.0
550	2476	2447	2470	2441	2471	2441	2456	17	4.7	0.0
600	2690	2650	2676	2645	2678	2645	2661	19	4.6	0.0
650	2919	2852	2886	2847	2890	2847	2868	24	4.6	0.0
700	3186	3049	3095	3045	3097	3045	3071	29	4.5	0.0
750	3501	3233	3294	3233	3294	3237	3264	34	4.5	0.0
800	3834	3384	3487	3402	3479	3413	3445	44	4.4	0.1

[8] This test-piece was heated significantly above the recommended use temperature described for BCR 724a and shows some net shrinkage in the first thermal cycle as a result.

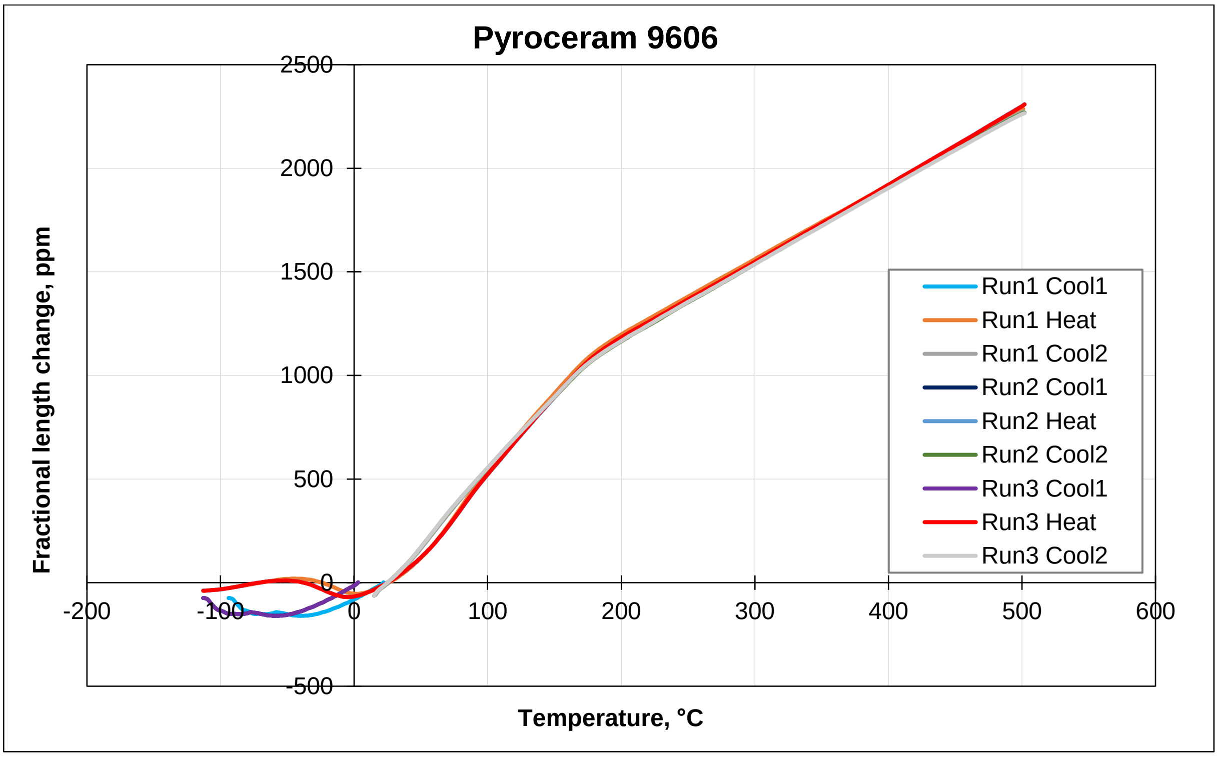


Figure 24: Linseis L75VS low-temperature dilatometer: Fractional length change as a function of temperature for a Pyroceram 9606 sample over three heating and cooling cycles

Table 20: Linseis L75VS low-temperature dilatometer: Fractional length change and mean expansion coefficient as a function of temperature of Pyroceram 9606

Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-32	1	0.3	0.0
-50	10	0	-0.1	0.0
0	-66	0	2.7	0.0
25	0	0	-	-
50	146	29	5.8	1.1
100	538	18	7.2	0.2
150	896	1	7.2	0.0
200	1174	10	6.7	0.1
250	1360	7	6.0	0.0
300	1542	6	5.6	0.0
350	1727	6	5.3	0.0
400	1913	8	5.1	0.0
450	2099	12	4.9	0.0
500	2282	21	4.8	0.0

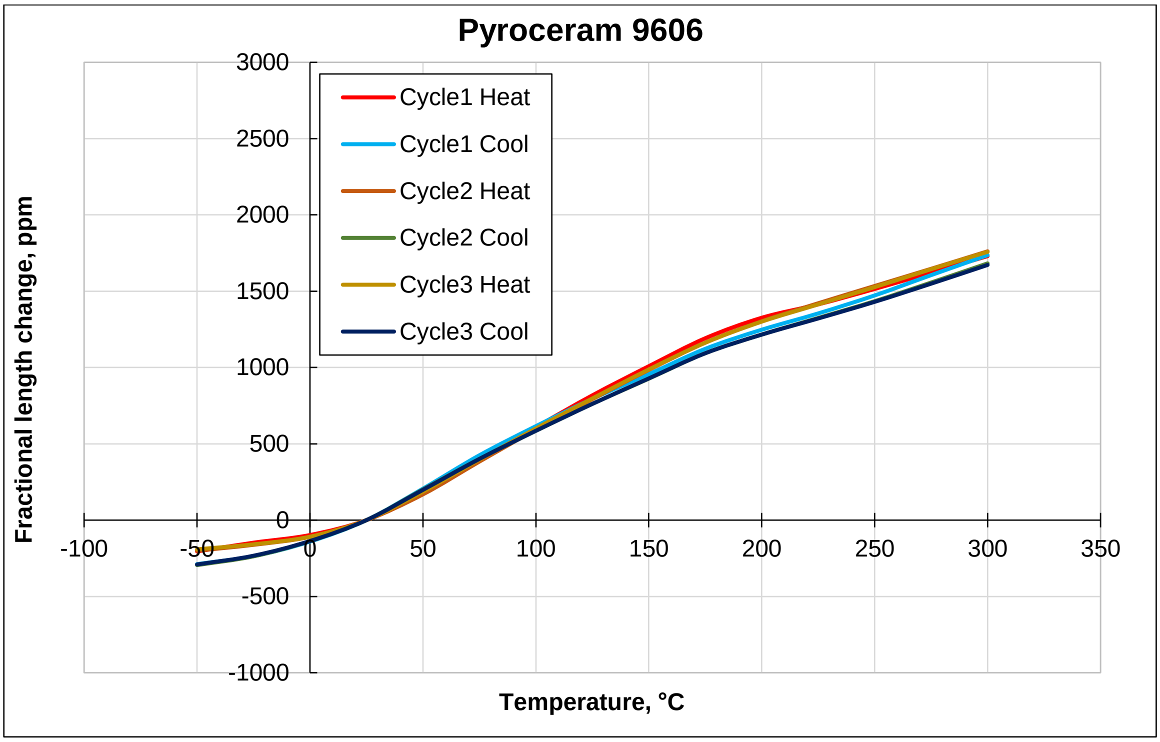


Figure 25: TMA: Fractional length change as a function of temperature for a Pyroceram 9606 sample over three heating and cooling cycles

Table 21: TMA: Fractional length change and mean expansion coefficient as a function of temperature of Pyroceram 9606

Temp., °C	Fractional length change from 25 °C, ppm								Exp. coeff. from 25 °C, 10^{-6} K^{1}	
	Cycle1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev.
-50	-205	-288	-200	-295	-191	-291	-244	56	3.3	-0.7
-25	-148	-236	-162	-237	-158	-233	-198	43	4.0	-0.9
0	-98	-141	-109	-137	-109	-137	-123	16	4.9	-0.7
25	0	0	0	0	0	0	0	0	-	-
50	172	206	169	201	186	198	188	15	7.5	0.6
75	387	425	384	401	396	401	395	8	7.9	0.2
100	608	616	593	585	601	585	591	7	7.9	0.1
125	818	795	793	762	797	762	778	19	7.8	0.2
150	1009	956	984	927	987	929	957	33	7.7	0.3
175	1192	1123	1163	1094	1163	1094	1128	40	7.5	0.3
200	1326	1248	1303	1216	1301	1216	1259	50	7.2	0.3
225	1414	1355	1421	1325	1414	1322	1370	54	6.9	0.3
250	1515	1472	1534	1435	1527	1430	1482	57	6.6	0.3
275	1623	1606	1647	1557	1645	1550	1600	53	6.4	0.2
300	1730	1736	1762	1683	1758	1672	1719	48	6.2	0.2

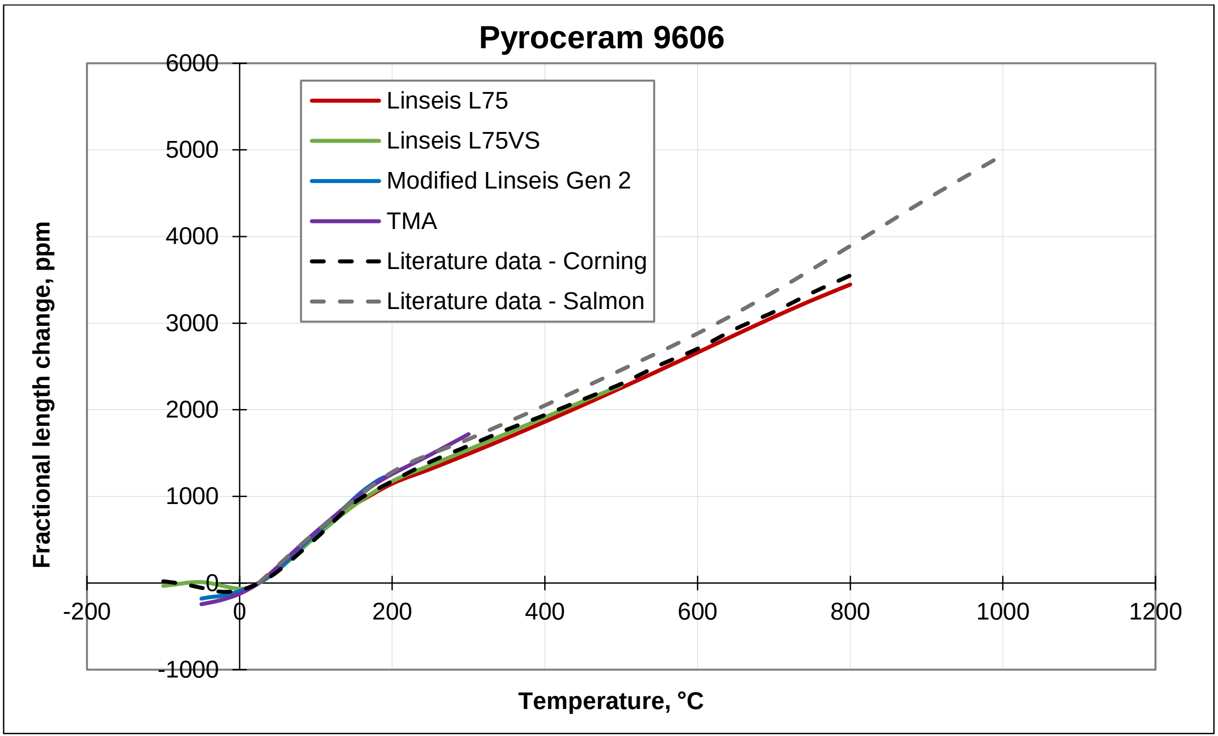


Figure 26: Comparison of the mean fractional length change as a function of temperature for a Pyroceram 9606 sample using the four systems and including literature data. [9, 10]

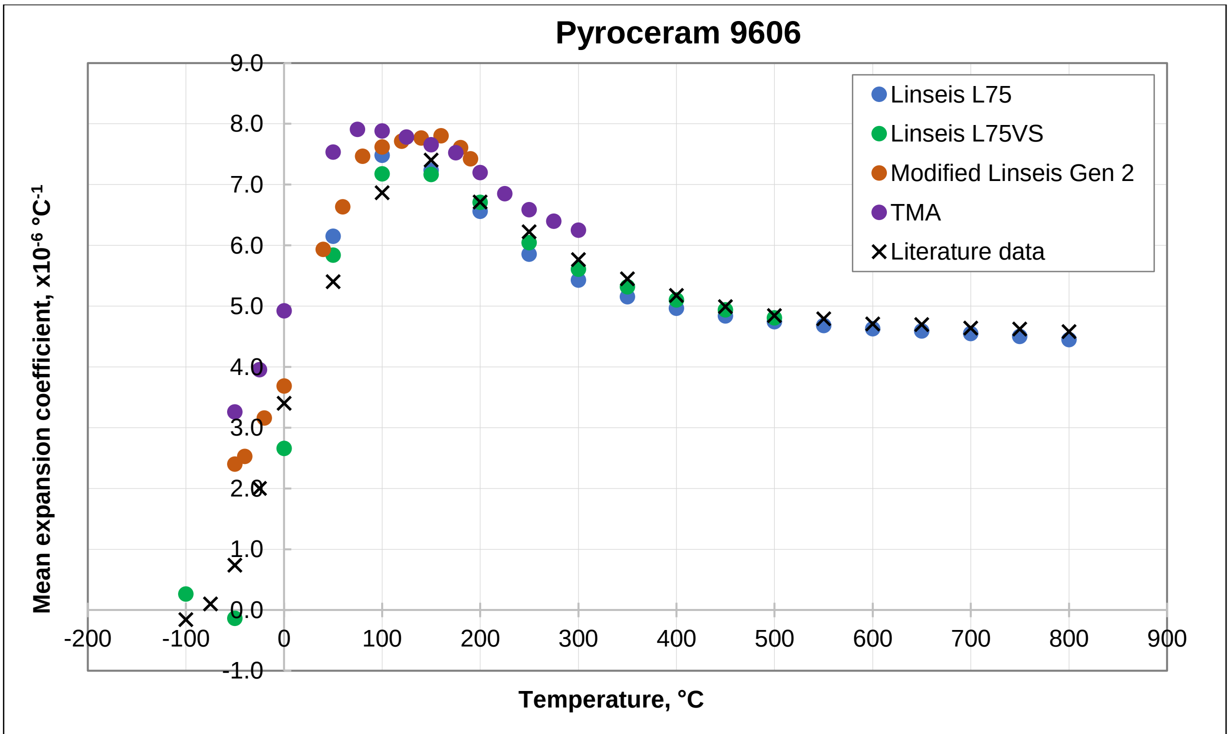


Figure 27: Comparison of the mean expansion coefficient as a function of temperature for a Pyroceram 9606 sample using the four systems and including literature data.

[9] Salmon D, Roebben G, Lamberty A, Brandt R. Certification of Thermal Conductivity and Thermal Diffusivity up to 1025 °K of a Ceramic Reference Material (BCR-724). EUR 21764 EN. 2007. JRC30964.
[10] Corning Glassworks data.

Table 22: Summary of averaged Pyroceram 9606 data for the four systems

Linseis L75

Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
25	0	0	-	-
50	154	21	6.1	0.8
100	561	12	7.5	0.2
150	904	6	7.2	0.0
200	1148	7	6.6	0.0
250	1317	9	5.9	0.0
300	1492	11	5.4	0.0
350	1674	12	5.2	0.0
400	1862	13	5.0	0.0
450	2055	14	4.8	0.0
500	2254	16	4.7	0.0
550	2456	17	4.7	0.0
600	2661	19	4.6	0.0
650	2868	24	4.6	0.0
700	3071	29	4.5	0.0
750	3264	34	4.5	0.0
800	3445	44	4.4	0.1

Linseis L75VS

Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-32	1	0.3	0.0
-50	10	0	-0.1	0.0
0	-66	0	2.7	0.0
25	0	0	-	-
50	146	29	5.8	1.1
100	538	18	7.2	0.2
150	896	1	7.2	0.0
200	1174	10	6.7	0.1
250	1360	7	6.0	0.0
300	1542	6	5.6	0.0
350	1727	6	5.3	0.0
400	1913	8	5.1	0.0
450	2099	12	4.9	0.0
500	2282	21	4.8	0.0

Modified Linseis Gen 2

Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-50	-180	32	2.4	-0.4
-40	-164	28	2.5	-0.4
-20	-142	18	3.2	-0.4
0	-92	13	3.7	-0.5
20	-22	8	4.5	-1.5
25	0	0	-	-
40	89	12	5.9	0.8
60	232	44	6.6	1.3
80	411	15	7.5	0.3
100	571	11	7.6	0.2
120	733	10	7.7	0.1
140	893	10	7.8	0.1
160	1053	10	7.8	0.1
180	1179	14	7.6	0.1
190	1225	18	7.4	0.1

TMA

Temp., °C	Fractional length change, ppm		Exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-50	-244	56	3.3	-0.7
-25	-198	43	4.0	-0.9
0	-123	16	4.9	-0.7
25	0	0	-	-
50	188	15	7.5	0.6
75	395	8	7.9	0.2
100	591	7	7.9	0.1
125	778	19	7.8	0.2
150	957	33	7.7	0.3
175	1128	40	7.5	0.3
200	1259	50	7.2	0.3
225	1370	54	6.9	0.3
250	1482	57	6.6	0.3
275	1600	53	6.4	0.2
300	1719	48	6.2	0.2

3.6 MOLYBDENUM

Moybdenum is a low expansion stable metal but has not been used as a certified reference material because it lacks resistance to oxidation. It has been used in this work for convenience because NIST data exist to very low temperatures. The source of the test material used was Goodfellow and is of 99.9% purity.

In Figure 32 and 33 a comparison is made between the fractional length and the mean expansion coefficients determined from a 25 °C origin.

Table 27 gives an overall summary of the averaged data for each instrument.

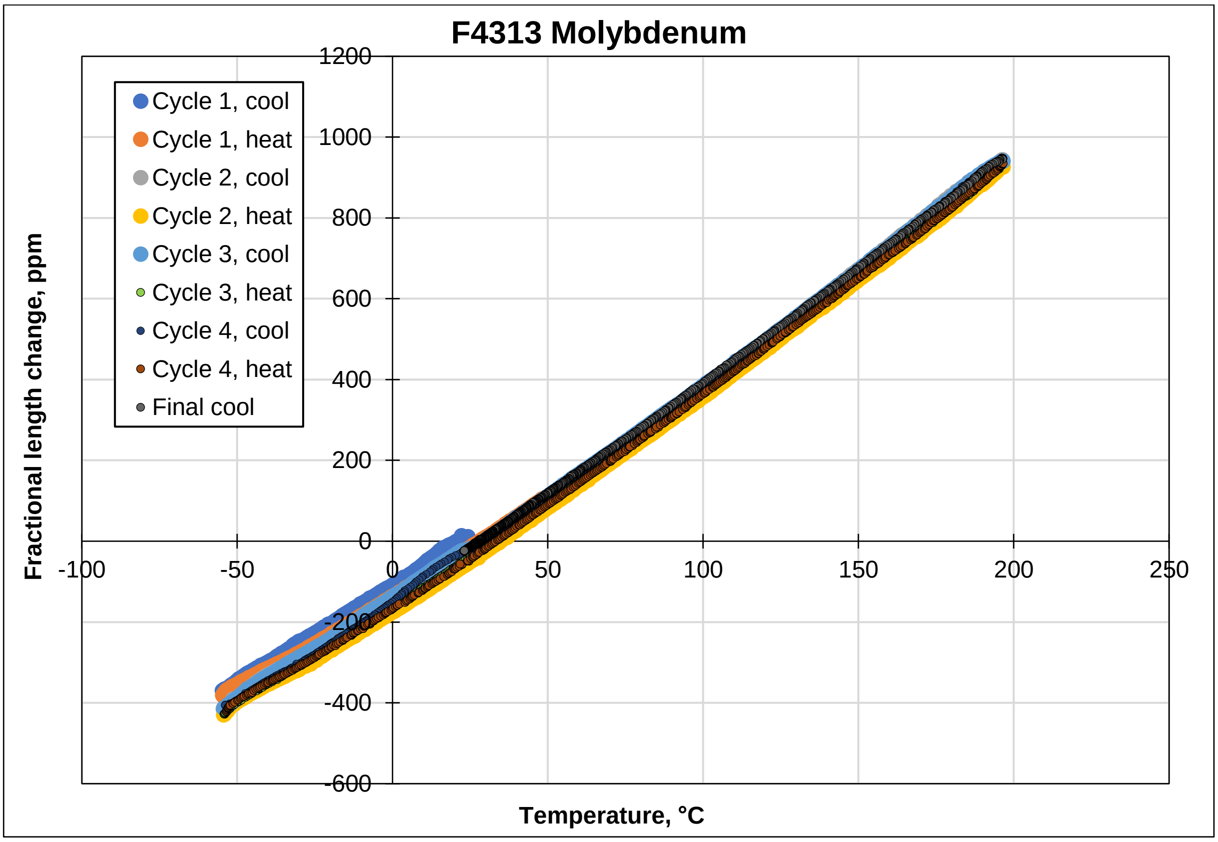


Figure 28: Modified Linseis Gen 2: Fractional length change as a function of temperature for a molybdenum sample over four heating and cooling cycles.

Table 23: Modified Linseis Gen 2: Fractional length change and mean expansion coefficient as a function of temperature of molybdenum.

Temp, °C	Fractional length change, ppm, from 20 °C											Expansion coeff. from 25 °C,10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Cycle 4		Final	Mean from 25 °C			
	Cool	Heat	Cool	Heat	Cool	Heat	Cool	Heat	cool	Mean	Std.dev.	Average	Std.dev.
-50	-356	-338	-260	-347	-364	-360	-375	-350	-	-343	36	4.6	-0.5
-40	-310	-298	-303	-303	-309	-313	-326	-305	-	-310	9	4.8	-0.1
-20	-216	-215	-222	-220	-218	-220	-236	-219	-	-223	7	4.9	-0.1
0	-120	-121	-129	-125	-114	-124	-132	-123	-	-125	6	5.0	-0.2
20	-14	-23	-19	-25	-11	-25	-18	-25	-	-21	5	4.1	-1.1
25	0	0	0	0	0	0	0	0	0	0	0	-	-
40	-	77	81	80	82	78	79	80	81	80	2	5.3	0.1
60	-	178	189	188	190	186	188	188	188	188	4	5.4	0.1
80	-	284	298	299	298	295	296	297	295	297	5	5.4	0.1
100	-	391	411	408	408	408	408	409	406	409	6	5.4	0.1
120	-	501	524	523	519	521	520	523	519	522	7	5.5	0.1
140	-	615	639	639	636	635	695	636	633	647	23	5.6	0.2
160	-	724	759	754	753	749	754	754	750	754	11	5.6	0.1
180	-	843	883	873	875	864	871	870	865	873	12	5.6	0.1
190	-	906	938	935	936	927	932	932	929	933	10	5.7	0.1

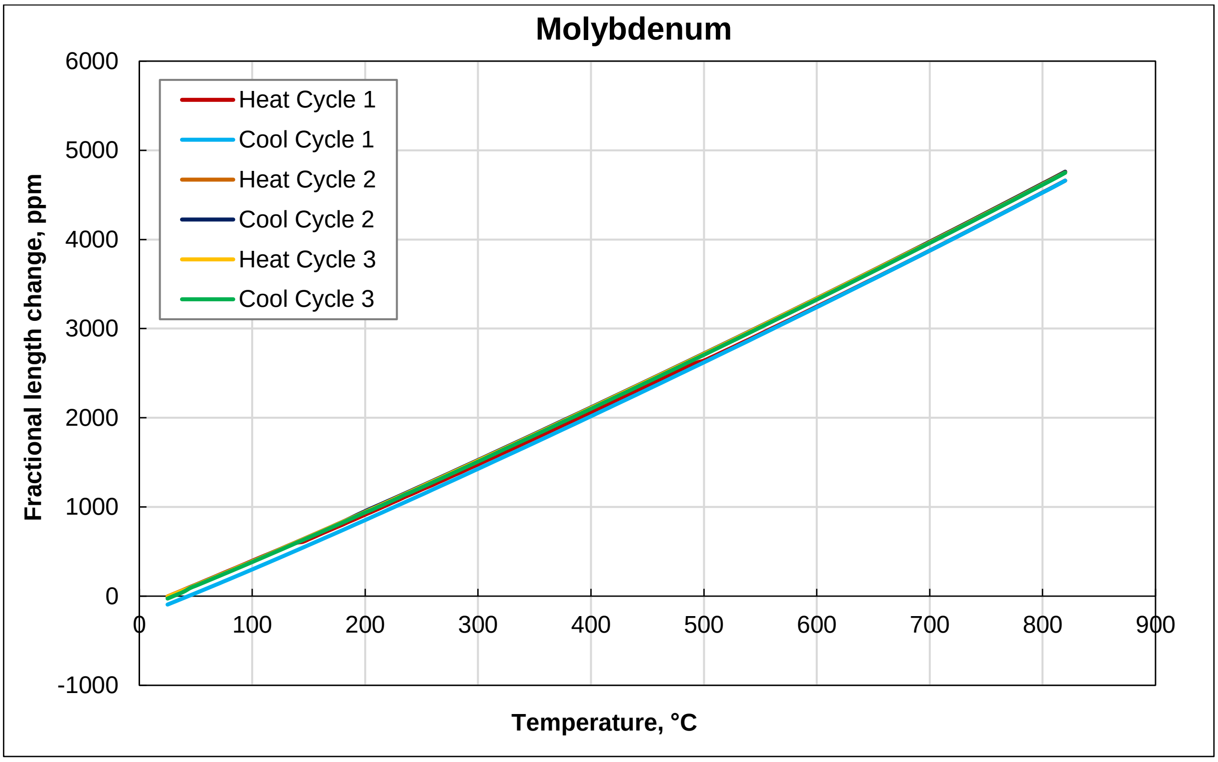


Figure 29: Linseis L75 high-temperature dilatometer: Fractional length change as a function of temperature for a molybdenum sample over three heating and cooling cycles.

Table 24: Linseis L75 high-temperature dilatometer: Fractional length change and mean expansion coefficient as a function of temperature of molybdenum.

Temp., °C	Fractional length change from 25 °C, ppm								Expansion coeff. from 25 °C, 10^{-6} K^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev
50	128	125	125	130	124	140	130	7	5.2	0.3
100	396	390	391	396	390	406	396	7	5.3	0.1
150	638	663	665	669	664	681	670	7	5.4	0.1
200	914	943	945	957	944	962	952	9	5.4	0.1
250	1195	1228	1230	1240	1230	1243	1236	7	5.5	0.0
300	1477	1518	1521	1530	1521	1532	1526	6	5.5	0.0
350	1769	1811	1815	1823	1813	1827	1820	7	5.6	0.0
400	2063	2108	2113	2120	2111	2124	2117	6	5.6	0.0
450	2364	2408	2414	2421	2413	2425	2418	6	5.7	0.0
500	2641	2712	2719	2724	2717	2729	2722	5	5.7	0.0
550	2940	3019	3028	3032	3025	3037	3031	5	5.8	0.0
600	3247	3330	3339	3343	3337	3349	3342	5	5.8	0.0
650	3558	3645	3655	3658	3651	3664	3657	6	5.9	0.0
700	3875	3964	3974	3977	3966	3983	3975	7	5.9	0.0
750	4199	4287	4299	4301	4290	4307	4299	7	5.9	0.0
800	4528	4616	4628	4630	4618	4636	4628	8	6.0	0.0

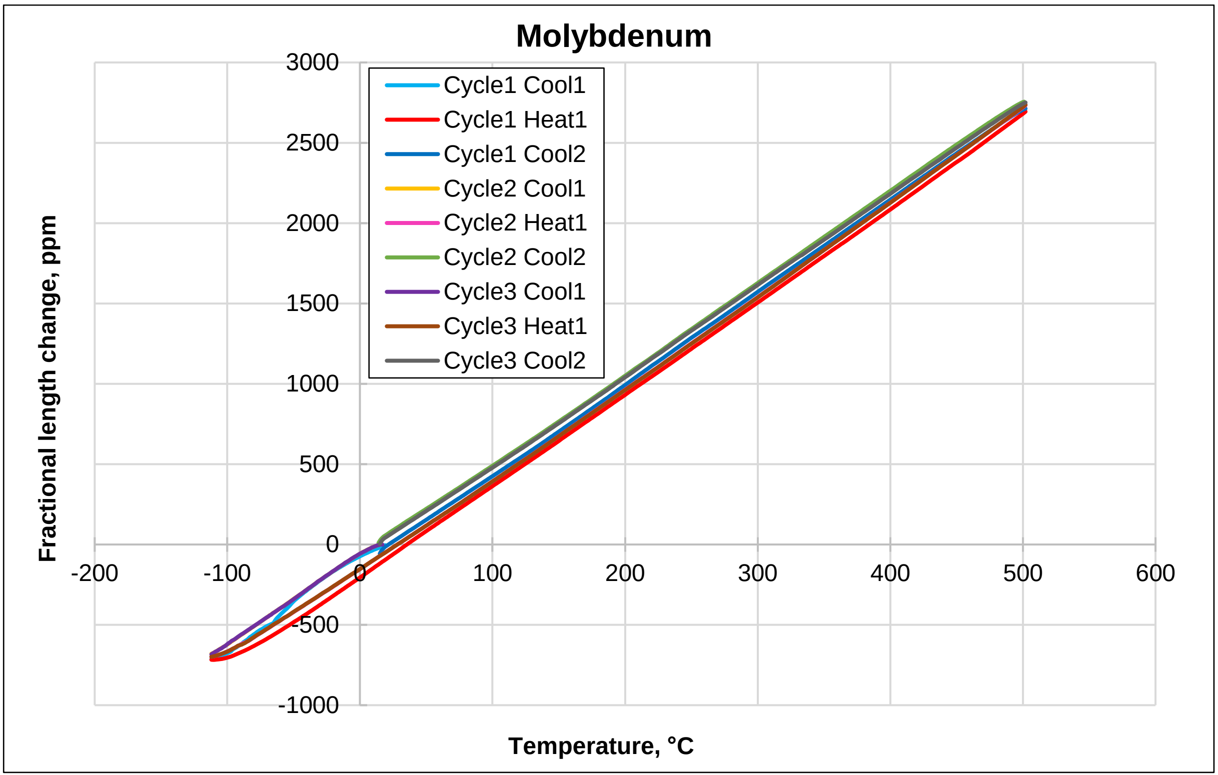


Figure 30: Linseis L75VS low-temperature dilatometer: Fractional length change as a function of temperature for a molybdenum sample over three heating and cooling cycles.

Table 25: Linseis L75VS Low-temperature dilatometer: Fractional length change and mean expansion coefficient as a function of temperature of molybdenum.

Temp., °C	Fractional length change, ppm		Mean exp. coeff. from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-647	3	5.2	0.0
-50	-409	12	5.5	-0.2
0	-139	5	5.6	-0.2
25	0	0	-	-
50	136	4	5.4	0.2
100	409	8	5.5	0.1
150	688	10	5.5	0.1
200	976	11	5.6	0.1
250	1265	10	5.6	0.0
300	1552	11	5.6	0.0
350	1841	14	5.7	0.0
400	2130	17	5.7	0.0
450	2422	22	5.7	0.1
500	2707	37	5.7	0.1

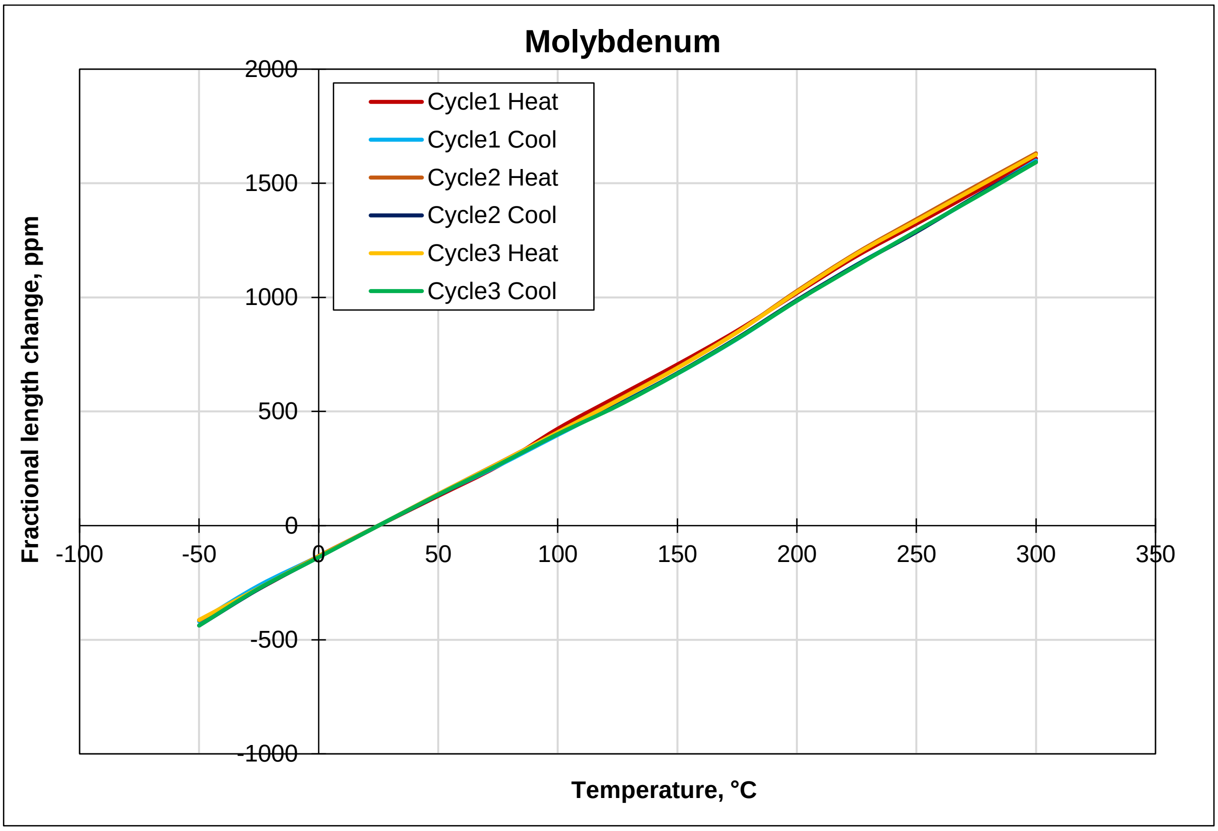


Figure 31: TMA: Fractional length change as a function of temperature for a molybdenum sample over three heating and cooling cycles.

Table 26: TMA: Fractional length change and mean expansion coefficient as a function of temperature of molybdenum.

Temp., °C	Fractional length change from 25 °C, ppm								Expansion coeff from 25 °C, 10^{-6} °C^{-1}	
	Cycle 1		Cycle 2		Cycle 3		Mean cycle 2 & 3			
	Heat	Cool	Heat	Cool	Heat	Cool	Mean	Std.dev.	Mean	Std.dev.
-50	-414	-420	-415	-438	-413	-438	-426	14	5.7	-0.2
-25	-277	-262	-276	-277	-274	-275	-276	1	5.5	0.0
0	-135	-134	-133	-138	-135	-139	-136	2	5.5	-0.1
25	0	0	0	0	0	0	0	0	-	-
50	130	135	138	134	138	135	136	2	5.5	0.1
75	261	262	272	267	271	266	269	3	5.4	0.1
100	425	397	410	402	409	401	406	5	5.4	0.1
125	566	530	550	528	548	525	538	13	5.4	0.1
150	706	665	692	668	691	665	679	14	5.4	0.1
175	854	817	848	821	847	818	834	16	5.6	0.1
200	1019	986	1028	988	1024	984	1006	23	5.7	0.1
225	1181	1143	1195	1142	1191	1139	1167	30	5.8	0.2
250	1322	1288	1343	1287	1336	1291	1314	29	5.8	0.1
275	1465	1441	1488	1444	1481	1439	1463	25	5.9	0.1
300	1608	1600	1631	1592	1625	1592	1610	21	5.9	0.1

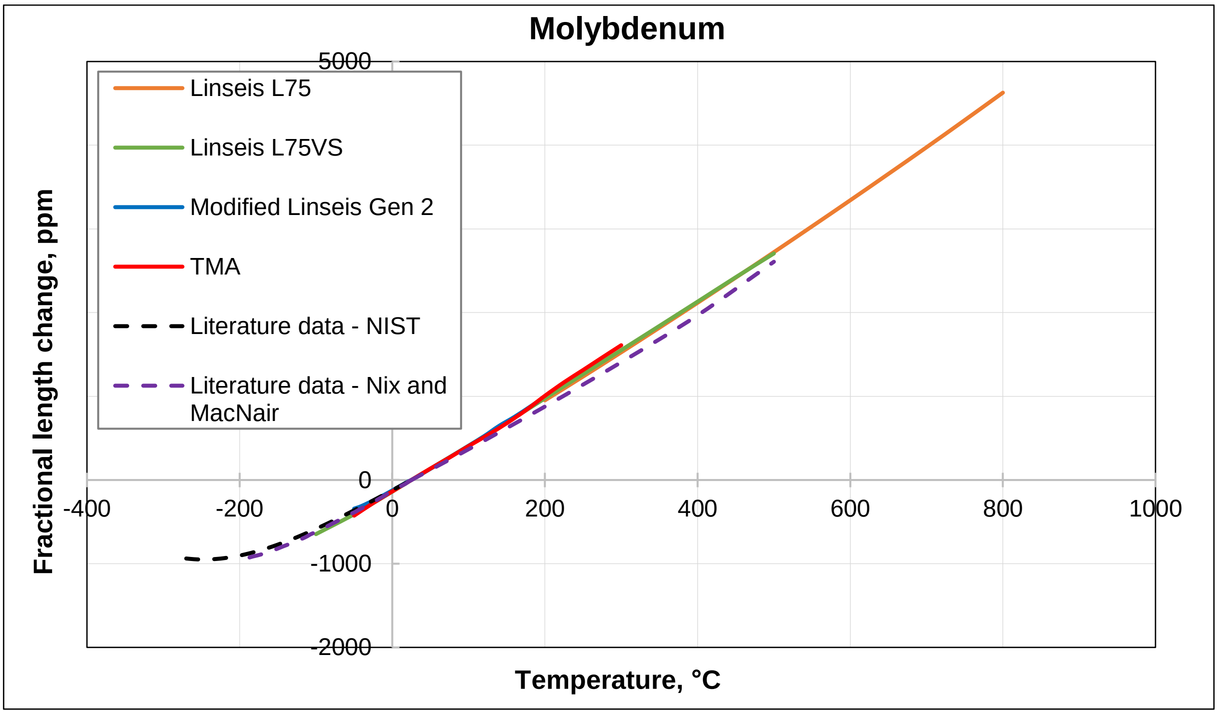
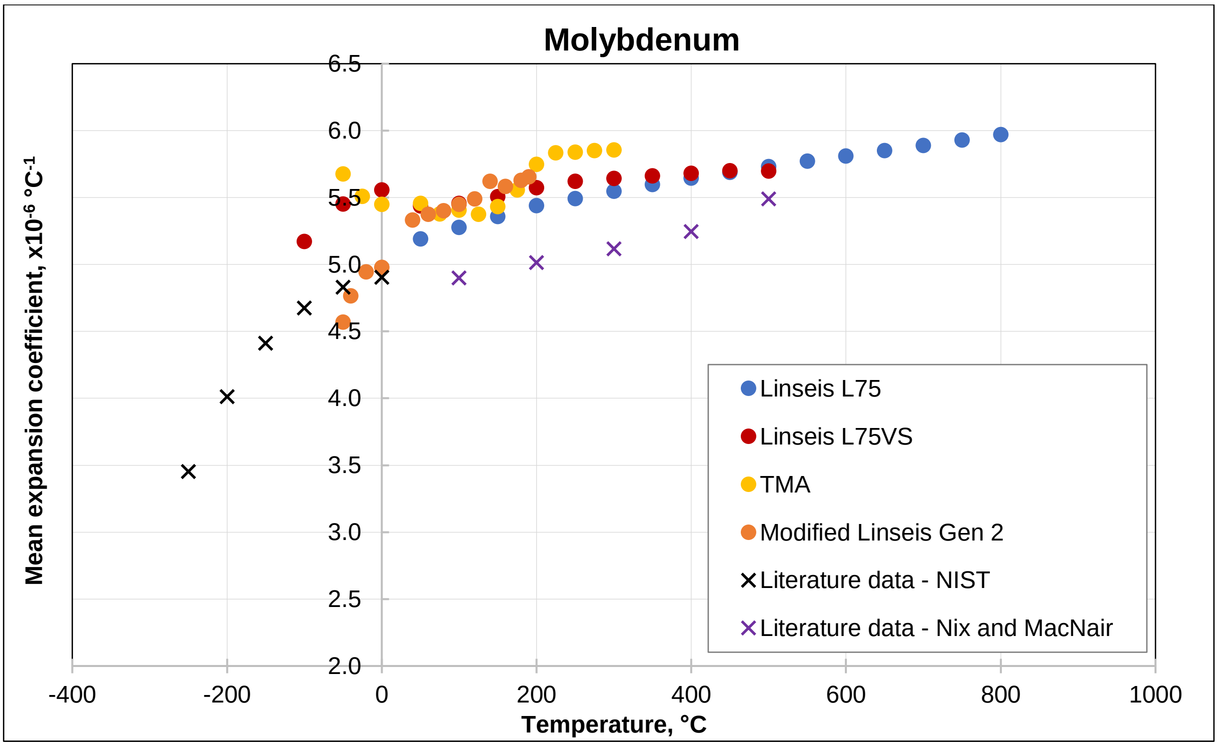


Figure 32: Comparison of the mean fractional length change as a function of temperature for a molybdenum sample using the four systems and including literature data.[11, 12]



[11] Nix, F. C. and MacNair, D. The Thermal Expansion of Pure Metals. II: Molybdenum, Palladium, Silver, Tantalum, Tungsten, Platinum, and Lead. Phys. Rev.,61,74 (1942)
[12] https://trc.nist.gov/cryogenics/materials/Molybdenum/Molybdenum_rev.htm

Figure 33: Comparison of the mean expansion coefficient as a function of temperature for a molybdenum sample using the four systems and including literature data.

Table 27: Summary of averaged molybdenum data for the four systems.

Linseis L75				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
50	130	7	5.2	0.3
100	396	7	5.3	0.1
150	670	7	5.4	0.1
200	952	9	5.4	0.1
250	1236	7	5.5	0.0
300	1526	6	5.5	0.0
350	1820	7	5.6	0.0
400	2117	6	5.6	0.0
450	2418	6	5.7	0.0
500	2722	5	5.7	0.0
550	3031	5	5.8	0.0
600	3342	5	5.8	0.0
650	3657	6	5.9	0.0
700	3975	7	5.9	0.0
750	4299	7	5.9	0.0
800	4628	8	6.0	0.0

Linseis L75VS				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-100	-647	3	5.2	0.0
-50	-409	12	5.5	-0.2
0	-139	5	5.6	-0.2
25	0	0	-	-
50	136	4	5.4	0.2
100	409	8	5.5	0.1
150	688	10	5.5	0.1
200	976	11	5.6	0.1
250	1265	10	5.6	0.0
300	1552	11	5.6	0.0
350	1841	14	5.7	0.0
400	2130	17	5.7	0.0
450	2422	22	5.7	0.1
500	2707	37	5.7	0.1

Modified Linseis Gen 2				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-50	-343	36	4.6	-0.5
-40	-310	9	4.8	-0.1
-20	-223	7	4.9	-0.1
0	-125	6	5.0	-0.2
20	-21	5	4.1	-1.1
25	0	0	-	-
40	80	2	5.3	0.1
60	188	4	5.4	0.1
80	297	5	5.4	0.1
100	409	6	5.4	0.1
120	522	7	5.5	0.1
140	647	23	5.6	0.2
160	754	11	5.6	0.1
180	873	12	5.6	0.1
190	933	10	5.7	0.1

TMA				
Temp., °C	Fractional length change, ppm		Exp. Coeff from 25 °C, 10^{-6} K^{-1}	
	Mean	Std.dev.	Mean	Std.dev.
-50	-426	14	5.7	-0.2
-25	-276	1	5.5	0.0
0	-136	2	5.5	-0.1
25	0	0	-	-
50	136	2	5.5	0.1
75	269	3	5.4	0.1
100	406	5	5.4	0.1
125	538	13	5.4	0.1
150	679	14	5.4	0.1
175	834	16	5.6	0.1
200	1006	23	5.7	0.1
225	1167	30	5.8	0.2
250	1314	29	5.8	0.1
275	1463	25	5.9	0.1
300	1610	21	5.9	0.1

4. DISCUSSION

4.1 COMPARISON OF INSTRUMENTS

Overall, there is a reasonably good agreement of Linseis L75, Modified Linseis Gen 2 and the new low temperature Linseis L75VS to the literature values for copper, platinum, Inconel 718 and Pyroceram 9606. The TMA results show an overall higher mean fractional length change for all materials measured using this instrument apart from platinum, where there is a good agreement. All four instruments produced a steeper slope in fractional length change in the case of molybdenum when compared to the literature data. Similar trend was found for stainless steel 316L when measured using Linseis L75, Linseis L75VS and TMA.

A summary of results for the new Linseis L75VS is given in Tables 28 in terms of fractional length change difference in result compared with reference or literature data, and in Table 29 in terms of percentage of fractional length change. The new instrument showed good agreement with literature data for platinum, copper, and Inconel 718. For Pyroceram 9606, there was a good agreement with Corning literature data above room temperature but not below. In the case of molybdenum, there is a relatively good agreement from -100 °C to about 50 °C above which the results deviated by up to 12 %. Similarly, stainless steel 316L, despite having very similar thermal expansion as copper, which showed a good agreement with literature values, the results are higher than expected.

4.2 MEASURMENT UNCERTAINTIES

Uncertainties in mechanical dilatometry can come from many sources, including:

- Mechanical stability of the test-piece in the apparatus during thermal cycling; avoidance of ‘jacking’, lateral ratchetting, and mechanical jogging or vibration from the operating environment;
- Accuracy of calibration of the transducer system in terms of converting test-piece movement into an expansion through the data logging system;
- Repeatability of thermal gradients along the length of measurement system;
- Test-piece thermocouple output calibration;
- Temperature differences along the length of the test-piece and between the test-piece and the measuring thermocouple; heat transfer, thermal conductivity of the test-piece and whether or not there is contact between thermocouple and test-piece are important factors;
- Consistency of temperature ramps; starting and reversing ramps leads to transient responses; ramps should be as slow as economically feasible to minimise these effects;
- Repeatability of baseline behaviour;
- Length of test-piece (usually negligible compared with the above factors).

Most of these factors are difficult or impossible to quantify, so probably the best approach is to look at the repeatability over numerous tests with reference materials. Experience with the L75 and L75 Gen 2 dilatometers suggests that provided that the test-piece gives repeatable behaviour, fractional length change overall consistencies of typically ±50 ppm on a 30 mm length of test-piece over a 100 °C temperature range and ±100 ppm over a 500 °C temperature range are achievable, corresponding to $\pm 0.5 \times 10^{-6}$ K^{-1} and $\pm 0.2 \times 10^{-6}$ K^{-1} respectively in mean expansion coefficients. Overall uncertainties (i.e., k = 2) are approximately double these figures.

Table 28: Difference in fractional length change between literature and measured data determined for the Linseis L75VS

Temp, °C	Difference in FLC from literature data, from 25 °C, ppm, (FLC_{ref}-$FLC_{measured}$)					
	Copper	Molybdenum	Stainless steel 316L	Platinum	Inconel 718	Pyroceram
-100	25	30	151	-36	-39	262
-50	17	23	129	-54	-53	119
0	-4	2	57	-13	-25	22
25	0	-9	13	0	0	0
50	20	-14	-37	9	33	-8
100	79	-27	-125	12	62	-4
150	128	-46	-195	10	70	3
200	145	-77	-277	7	82	0
250	157	-113	-357	1	98	3
300	155	-144	-403	-12	120	3
350	154	-169	-418	-25	139	2
400	153	-178	-412	-43	146	1
450	152	-161	-429	-61	141	1
500	210	-93	-451	-121	111	1

Table 29: Difference in % fractional length change between literature and measured data determined for the Linseis L75VS

Temp, °C	Difference in % FLC from literature data, from 25 °C, %, ((FLC_{ref}-$FLC_{measured}$)/FLC_{ref}*100)					
	Copper	Molybdenum	Stainless steel 316L	Platinum	Inconel 718	Pyroceram
-100	-1.3	-4.8	-7.4	-3.5	-2.6	262.1
-50	-1.4	-5.9	-10.1	-8.4	-5.6	118.9
0	0.8	-1.3	-11.3	-5.8	-7.9	21.9
25	-	-	-	-	-	-
50	4.7	-11.9	-12.5	-3.8	-10.8	-8.1
100	6.0	-7.0	-11.4	-1.8	-6.5	-4.5
150	5.8	-7.2	-10.3	-0.9	-4.2	3.2
200	4.7	-8.6	-10.1	-0.4	-3.5	0.1
250	3.9	-9.8	-9.9	0.0	-3.2	2.9
300	3.1	-10.2	-9.0	0.5	-3.2	2.7
350	2.6	-10.1	-7.7	0.8	-3.1	2.4
400	2.3	-9.1	-6.5	1.2	-2.8	1.4
450	2.0	-7.1	-5.8	1.5	-2.3	1.0
500	2.4	-3.6	-5.4	2.6	-1.6	0.8

At the time of writing this report overall uncertainties for the new equipment cannot yet be provided; work is in progress to develop and refine the temperature calibration and measurement procedure, followed by determination of uncertainty budgets for the equipment. In addition, NPL has procured a certified reference material of single-crystal silicon suitable for cryogenic temperatures that will be used as a certified reference material for below room-temperature measurements in the future.