PURDUE UNIVERSITY
School of Materials Engineering

Effects of Thermal Treatments on the Microstructural and Mechanical Properties of Alloy and Tool Steels

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The aim of this study is to characterize heat-treated 52100, A2, M2, and M50 steels to understand the effects of the heat treatment on the distribution of alloying elements, morphology of the carbides, and properties of the bulk matrix to connect those characteristics with mechanical behavior. To achieve these goals, metallographic microscopy, scanning electron microscopy, electron dispersive microscopy, X-ray diffraction, and Rockwell hardness testing were used to understand and depict the changes on a sub-grain level within the alloys that are being examined. Microscopy was used to understand the changes that occur in microstructure and morphology after the heat treatment. Composition analysis provided insight into the ways the carbides change compositionally when heat treated. The carbides tend to become enriched with alloying elements after heat treatment. X-ray diffraction identified the stoichiometry of the carbides, verifying and substantiating the composition analysis. Hardness testing quantified the strengthening effect that the heat treatment had on the alloys and verified the effect of the change in carbide morphology. The applied heat treatment caused the formation of MC, M_2C, and M_6C carbides. The average change in hardness of the successfully heat-treated samples was 215.95% ± 32.58%. Carbide volume fraction decreased after heat treatment for an average decrease in volume fraction of 69.34% ± 4.69%.

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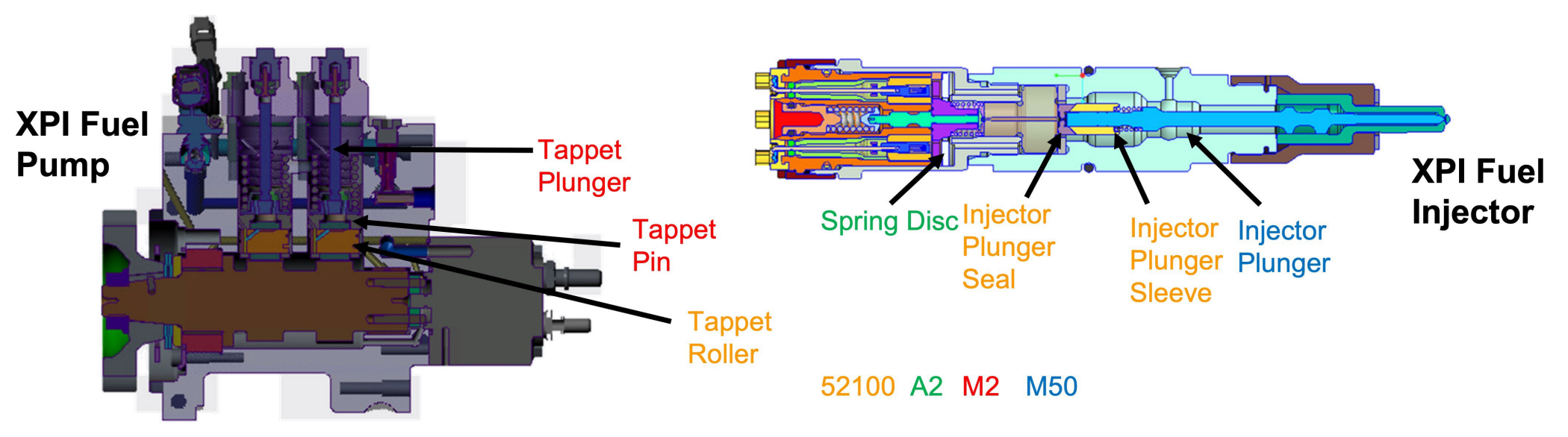
Introduction



- Steels used in high speed and high stress applications
- Wear resistance and fracture toughness are emphasized
- Thermal treatment applied to meet strength specifications
- Investigating the relationship between carbide morphology, composition, and strength
- Figure above shows example parts where the alloys are used

Materials and Methods

Materials

- 52100: high chromium carbon alloy steel
- A2: air-hardening cold work tool steel
- M2: high-speed molybdenum tool steel
- M50: tungsten-molybdenum high-speed steel

Grade	C	Mn	Si	Cr	Ni	Mo	W	V	P	S
52100	0.98 1.10	0.25 0.45	0.15 0.35	1.30 1.60	-	-	-	-	0.025 max	0.025 max
M2	0.78 0.88	0.15 0.40	0.20 0.45	3.75 4.50	0.30 max	4.50 5.50	5.50 6.75	1.75 2.20	0.03 max	0.03 max
M50	0.78 0.88	0.15 0.45	0.20 0.60	3.75 4.50	0.30 max	3.90 4.75	-	0.80 1.25	0.03 max	0.03 max
A2	0.95 1.05	1.00 max	0.50 max	4.75 5.50	0.30 max	0.90 1.40	-	0.15 0.50	0.03 max	0.03 max

Carbide Volume Analysis Grid Method

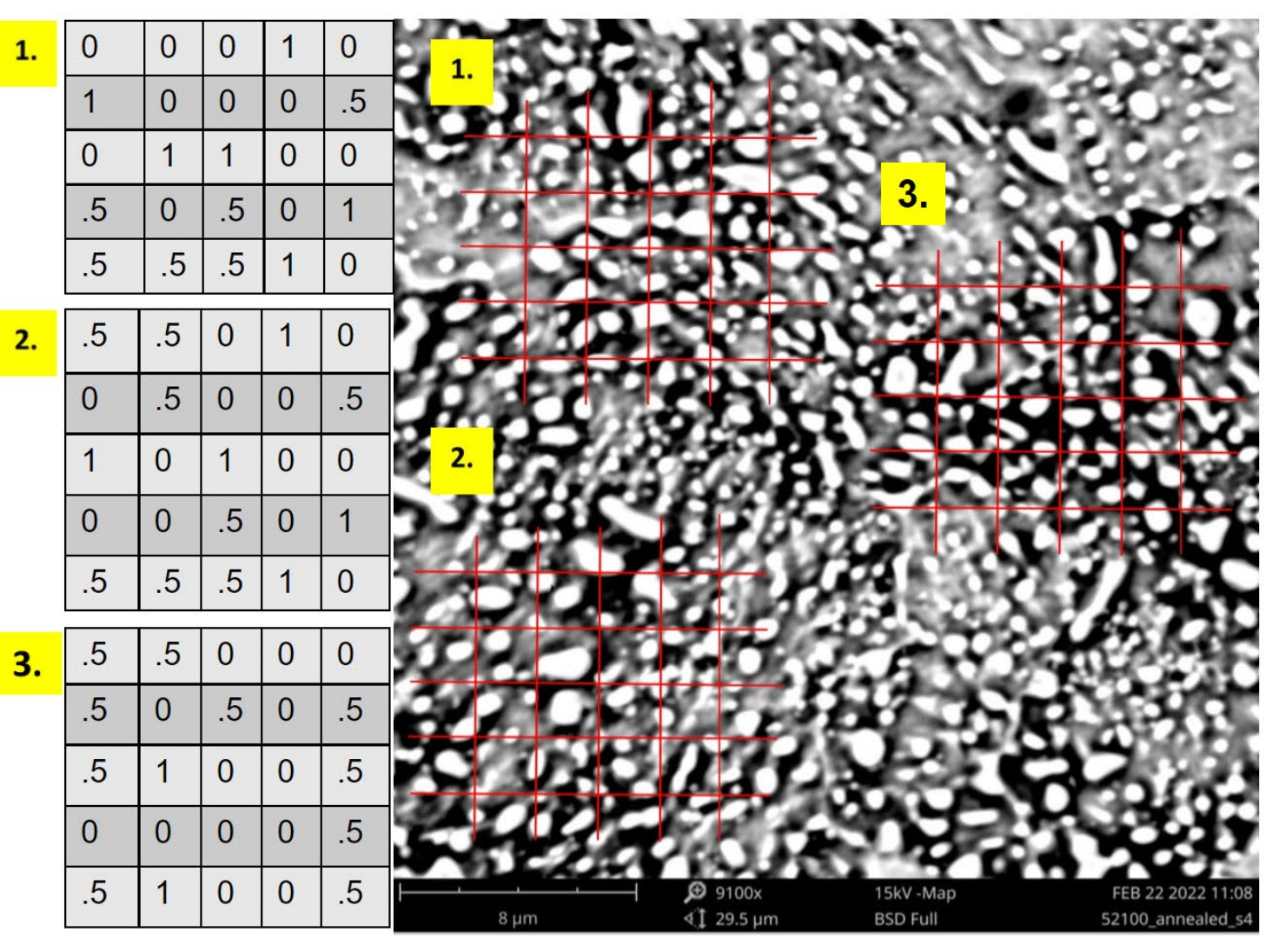
Grid Rules:

- Grid spacing should be approximately 2x size of average carbide size
- 5 X 5 grid, three grids per image
- Three images for each alloy (9 grids)
- Center = 1, Edge = .5, No contact = 0
- Volume fraction = Sum / (75*3)

Ex: (9+9+7) / 75 = .33

- $\sigma = \mathrm{SQRT}\left\{(V_V/P_T)(1-V_V)\right\}$.(1)

Reference: ASTM Int. (2019) ASTM E562-19, Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count



1.

0	0	0	1	0
1	0	0	0	.5
0	1	1	0	0
.5	0	.5	0	1
.5	.5	.5	1	0

2.

.5	.5	0	1	0
0	.5	0	0	.5
1	0	1	0	0
0	0	.5	0	1
.5	.5	.5	1	0

3.

.5	.5	0	0	0
.5	0	.5	0	.5
.5	1	0	0	.5
0	0	0	0	.5
.5	1	0	0	.5

- Optical metallography used to examine microstructure on a granular level
- EDS used to obtain semi-quantitative data on the surface compositions
- Rockwell Hardness testing was performed on each alloy to compare the change in hardness from the baseline heat treatment to annealing

X-Ray Diffraction

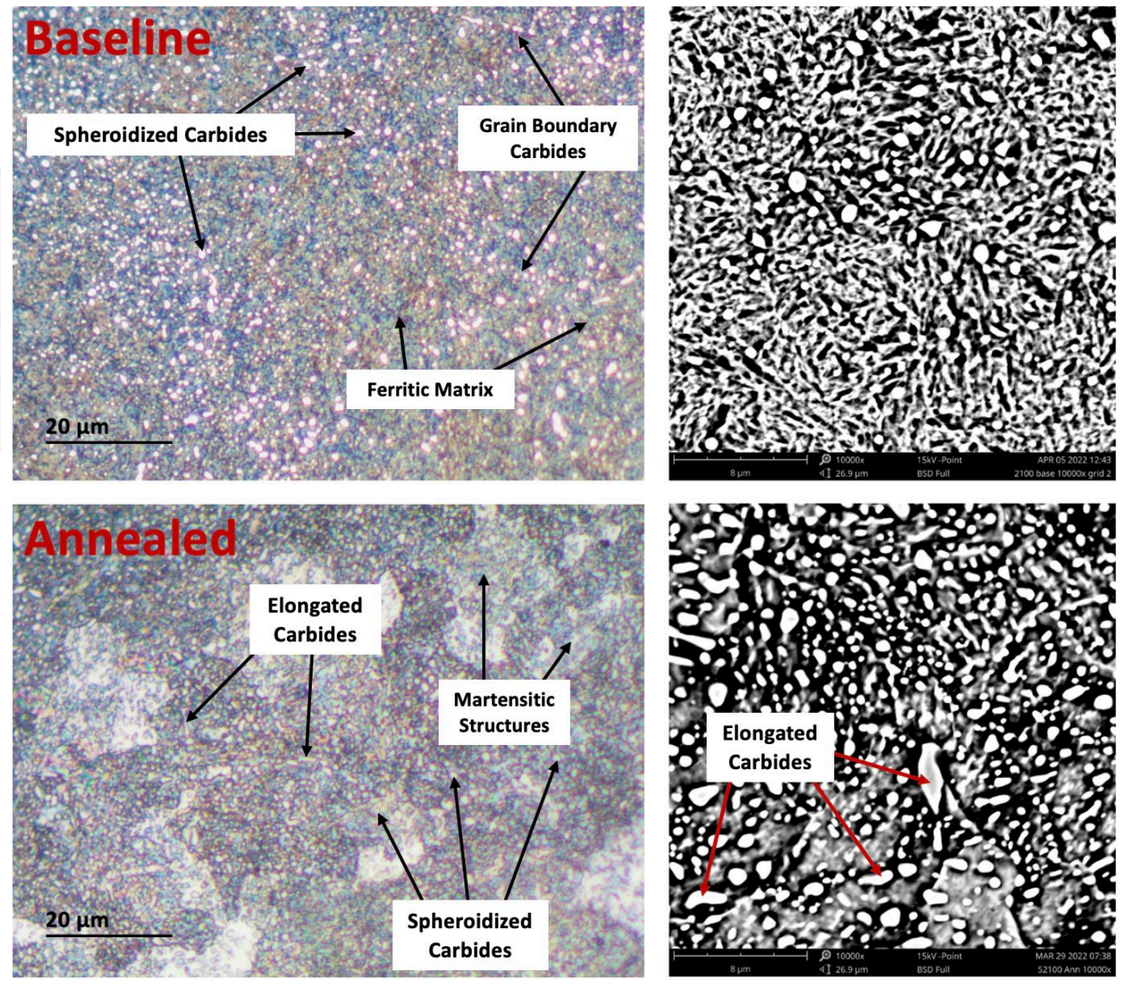
$$n\lambda = 2d\sin\theta \ (Bragg's\ Law) \qquad d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}}$$

- Formulae above were used to find characteristic peaks in XRD analysis.

Results

52100

Treatment	Spot Type (wt. %, avg.)	Large Carbide	Small Carbide	Matrix
Annealed	Cr	4.69	0.33	0.48
Baseline	Cr	7.94	2.24	0.66



Baseline

- Mainly composed of spheroidized carbides in a ferrite matrix
- Heavier carbide distribution around grain boundaries

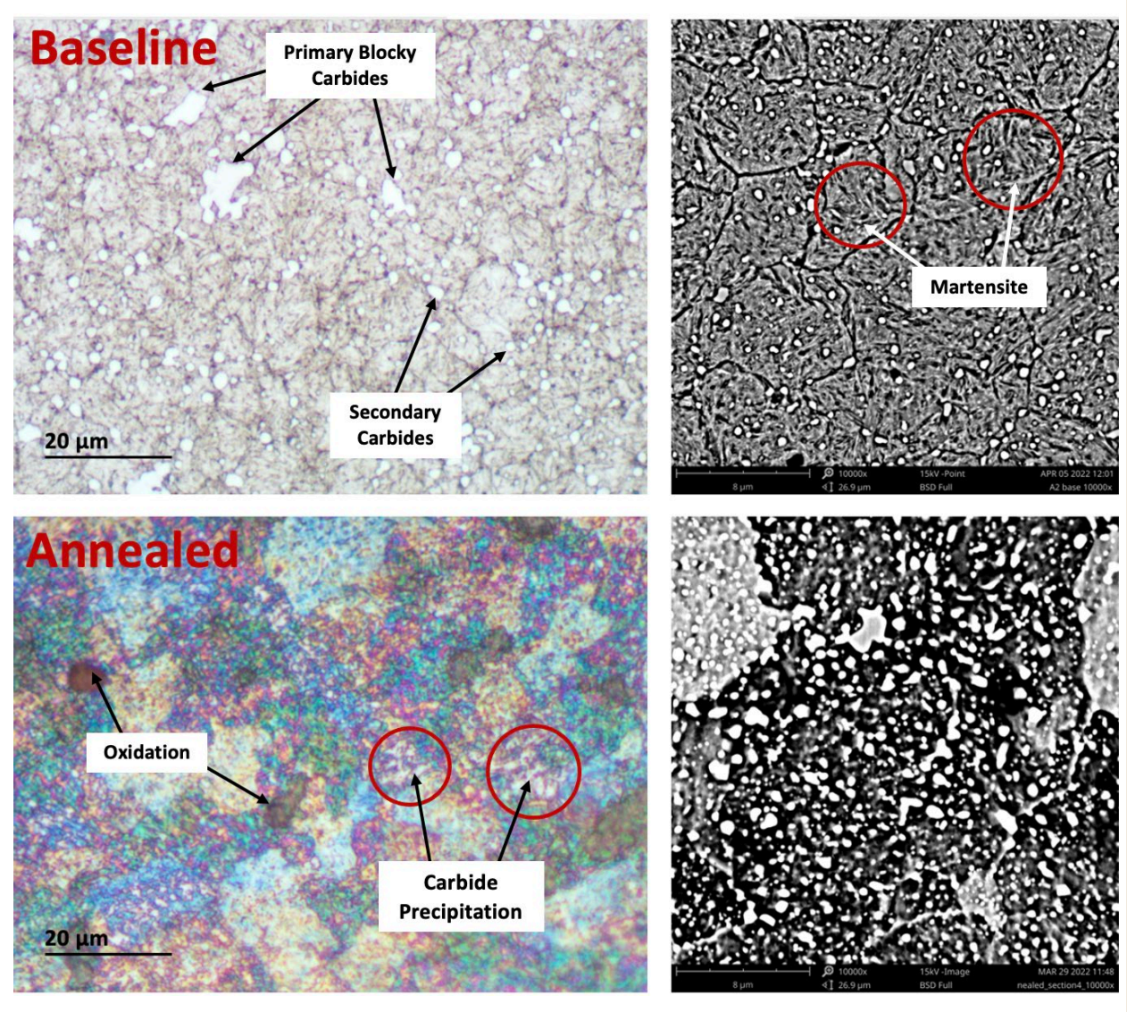
Annealed

Increased:

- Heterogeneity of carbide morphology
- Number of elongated carbides
- Carbide population
- Homogeneity of carbide distribution

A2

Treatment	Spot Type (wt. %, avg.)	Large Carbide	Small Carbide	Matrix
Annealed	Cr	25.12	10.84	3.29
Baseline	Cr	29.65	10.72	4.26
Annealed	Mo	5.22	4.79	0
Baseline	Mo	4.72	0	0
Annealed	V	1.53	0	0
Baseline	V	2.50	0.45	0



Baseline

- Fully martensitic matrix
- Distinct primary blocky carbides
- Predominantly populated with secondary carbides

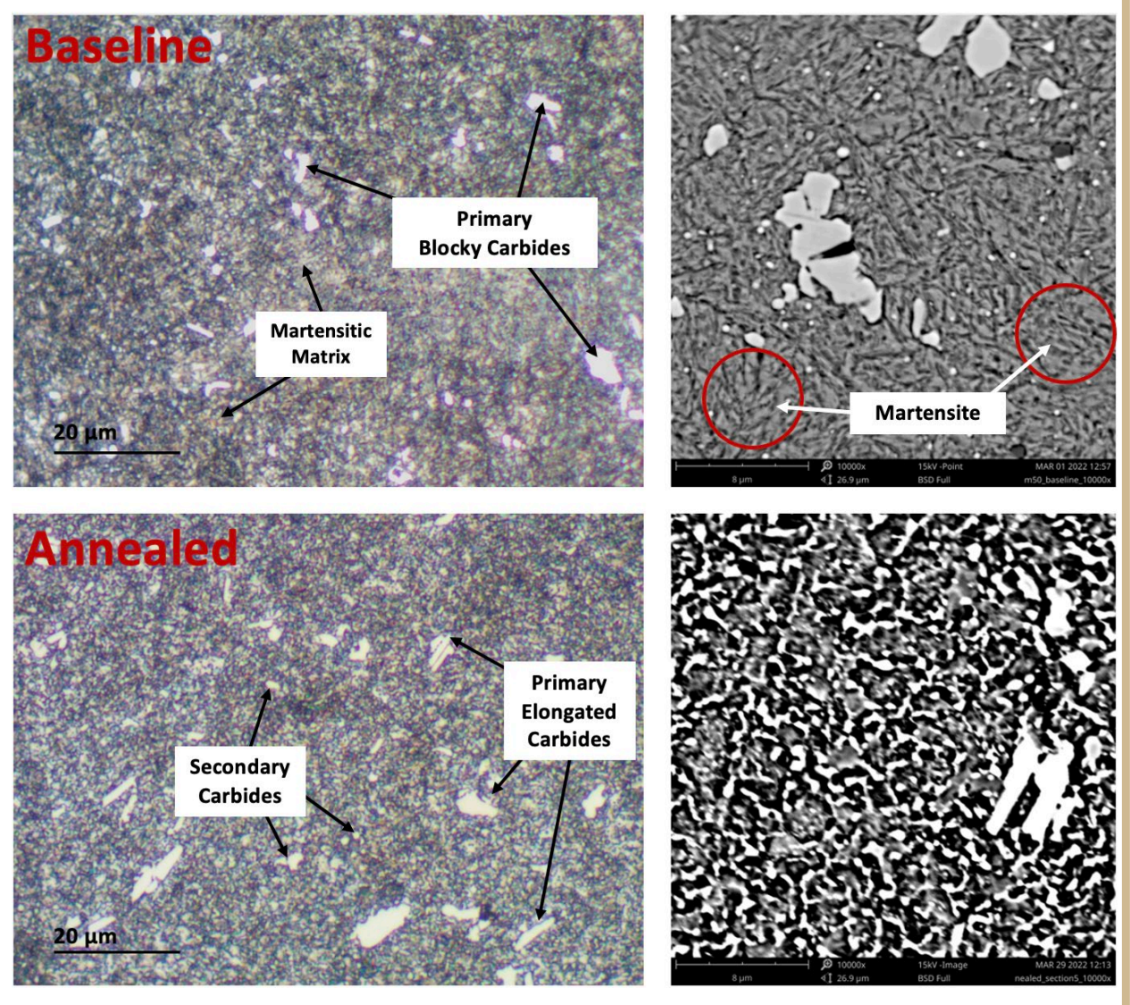
Annealed

- Precipitation of very fine carbides throughout matrix
- Reduced number or large carbides
- Increase in total carbide population
- Volume fraction

Results

M50

	Spot Type (wt %, avg)	Large Carbide	Small Carbide	Matrix
Annealed	Cr	11.09	11.25	3.91
Baseline	Cr	11.77	4.01	4.41
Annealed	Mn	0	0	0
Baseline	Mn	0	19.79	0
Annealed	Mo	56.23	7.34	4.65
Baseline	Mo	59.16	11.24	6.96
Annealed	V	13.00	0	0
Baseline	V	10.87	0	3.84



Baseline

- Blocky carbides of varying sizes
- Fine carbides seen scattered throughout martensitic matrix

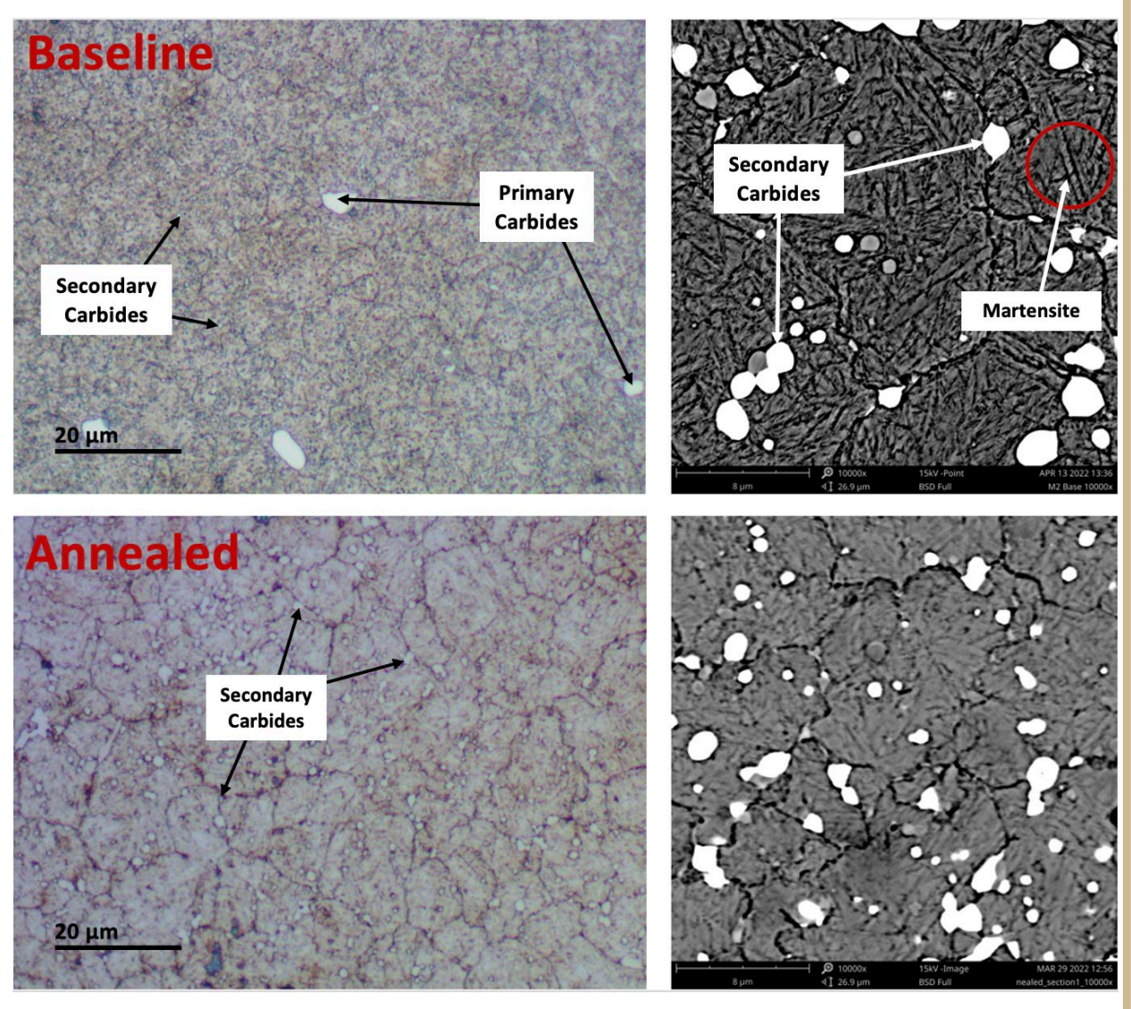
Annealed

Increased:

- Carbide distribution homogeneity
- Morphology homogeneity
- Number of elongated carbides
- Carbide population
- Carbide volume fraction

M2

	Spot Type (wt. %, avg.)	Large Carbide	Small Carbide	Matrix
Annealed	Cr	3.74	4.09	3.23
Baseline	Cr	3.72	3.95	4.38
Annealed	Mo	9.57	3.72	2.38
Baseline	Mo	21.52	15.06	1.44
Annealed	W	12.09	2.89	0.60
Baseline	W	30.25	19.93	0
Annealed	V	0.97	0.45	0.79
Baseline	V	2.60	8.29	0

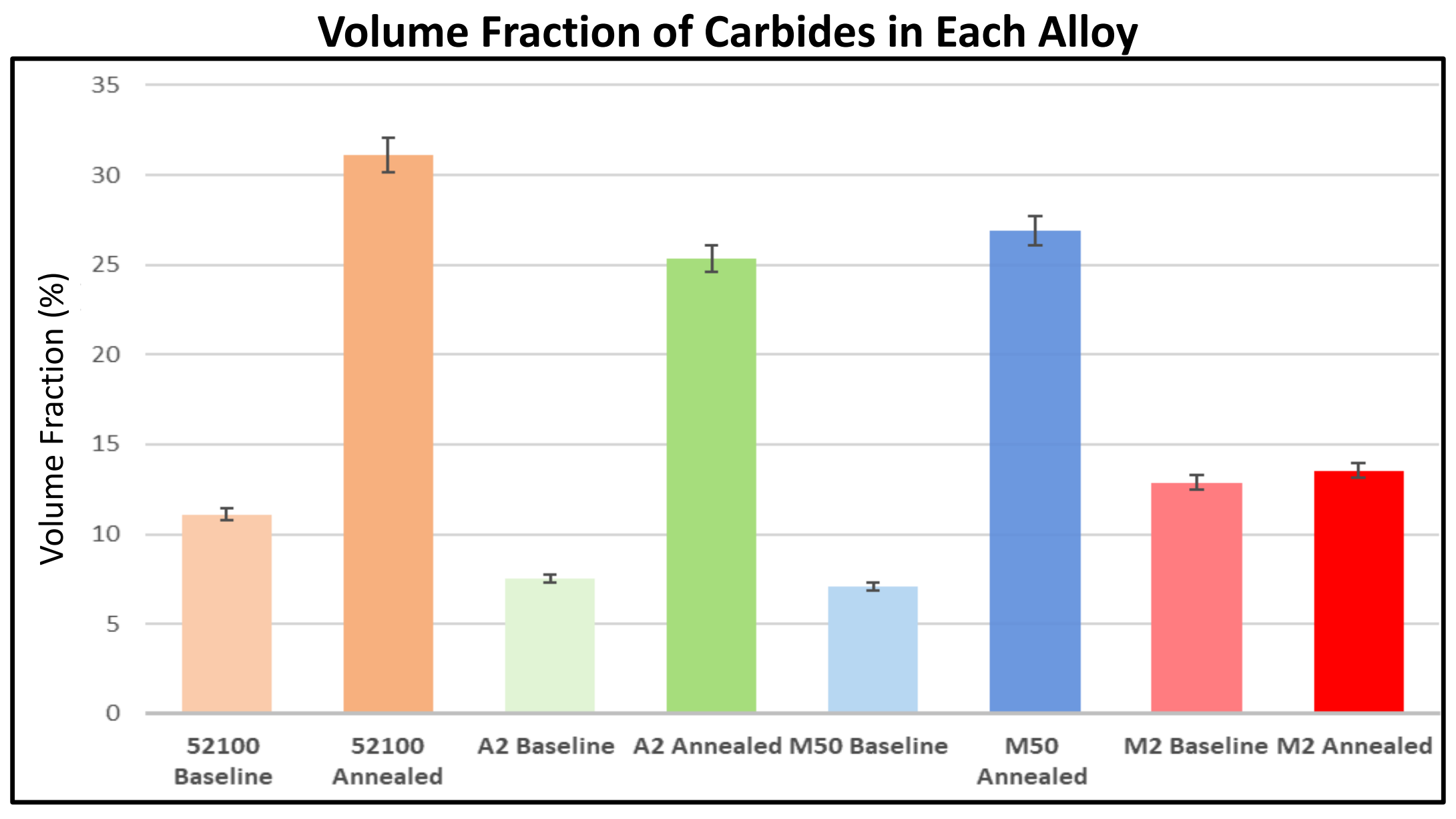


Baseline & Annealed exhibited similar microstructural features:

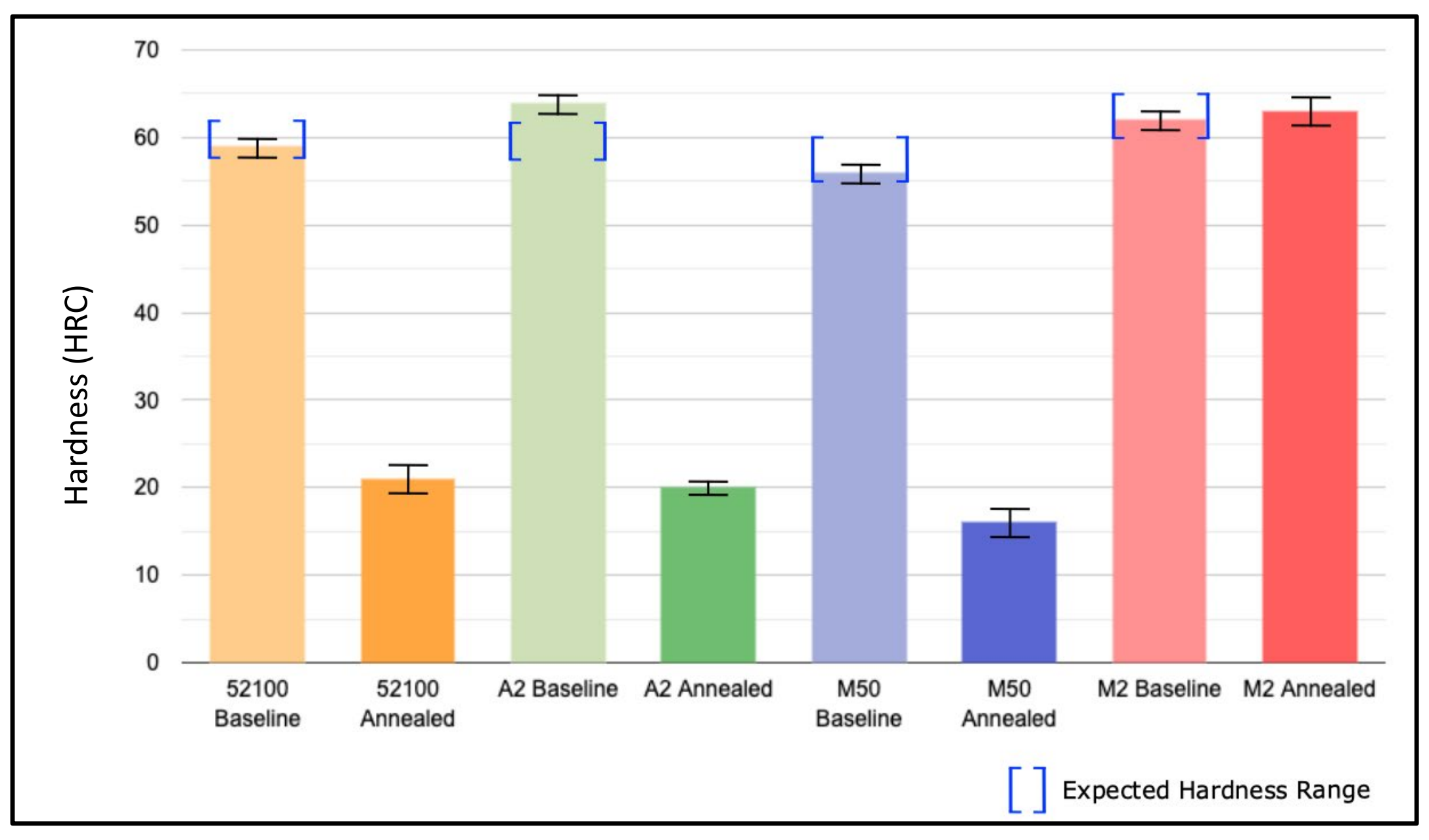
- High presence of needle-type martensite precipitation
- Moderate distribution of secondary carbides
- Significant carbide precipitation at grain boundaries

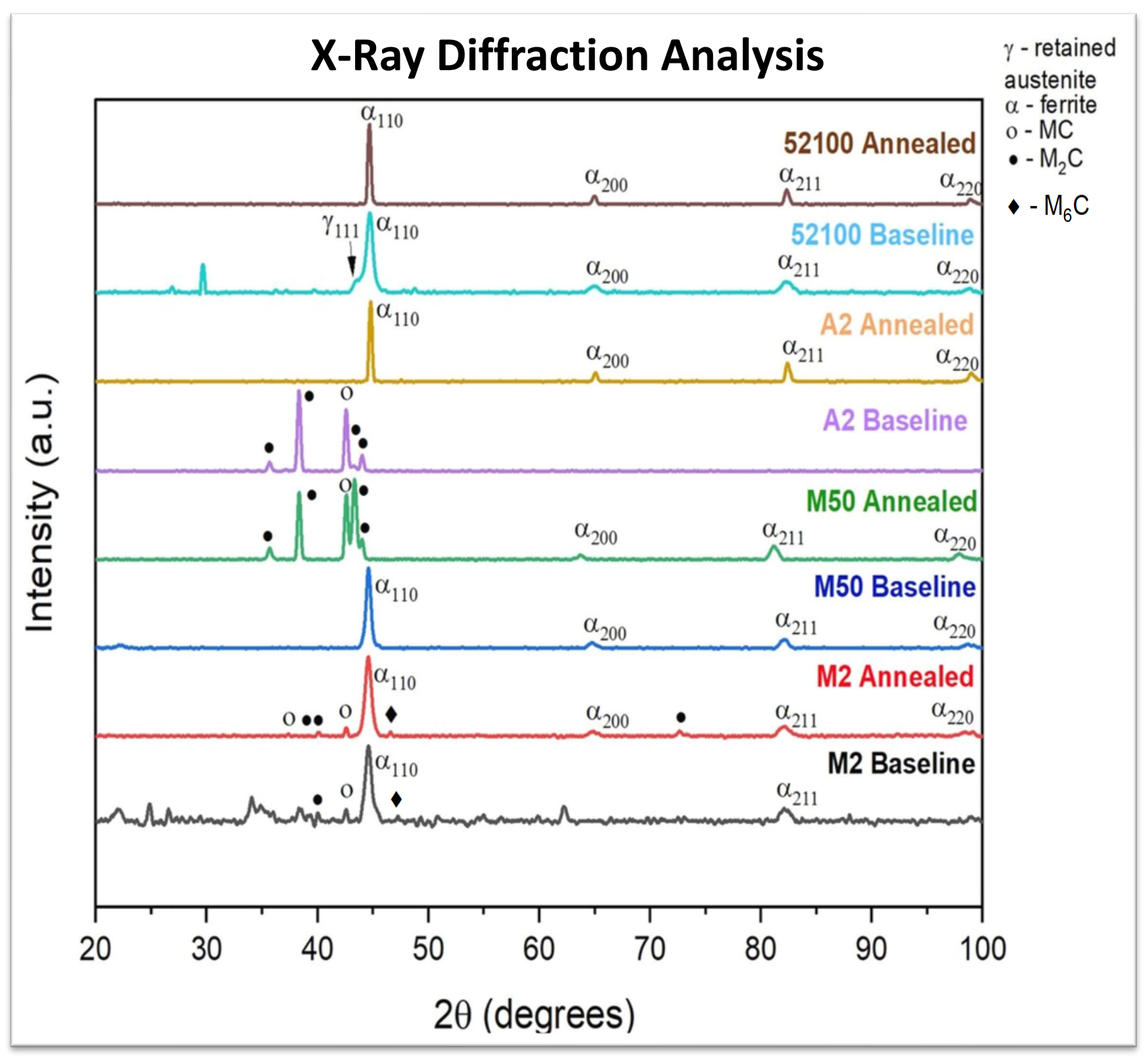
Baseline

- Large primary carbides more prominent


Volume Fraction of Carbides in Each Alloy

Hardness Comparison of Each Alloy




X-Ray Diffraction Analysis

- MC, M_2C, and M_6C carbides found.
- Retained austenite in 52100 Baseline.

Discussion

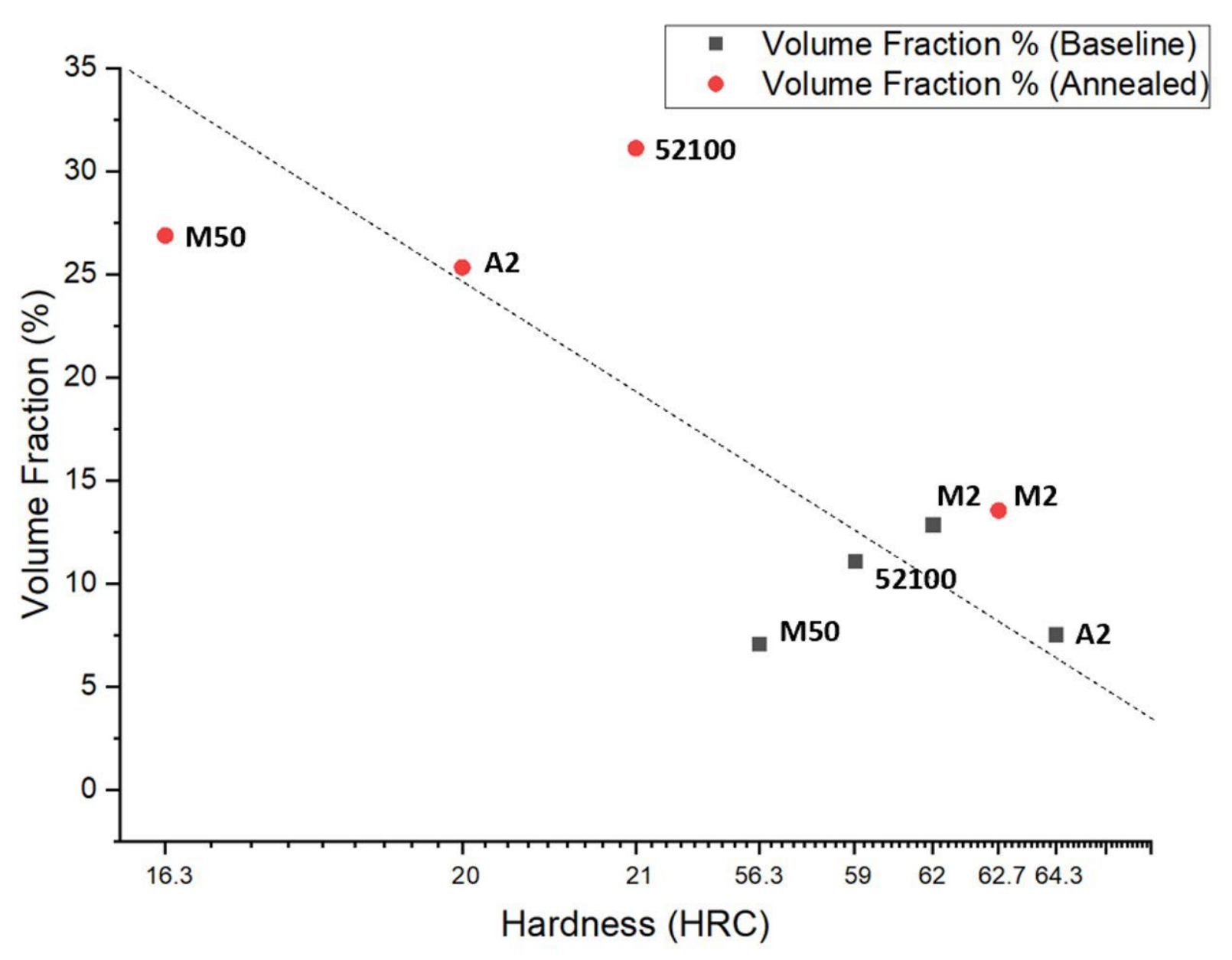
Microstructural Analysis

- Annealing 52100, A2, and M50 showed an increase in carbide population and distribution homogeneity and therefore the carbide volume fraction increased.
  - Dispersed precipitation of carbides produces precipitation hardening effects – however recrystallization hardening aids in reducing hardness
- Annealed 52100 and M50 samples exhibited a significant change in carbide morphology, from blockier or larger in size to smaller and more elongated in appearance
- Partially spheroidized carbides in the annealed 52100, A2, and M50 likely contributed to the decrease in strength in comparison to their baseline condition
  - Lack of orientation of elongated carbides and morphology effects provide to the isotropic properties and elevate the baseline material's hardness
- Martensite is a hard, brittle phase, so an increase in strength and hardness is expected with an increased content of martensite post annealing
- Ferritic and martensitic matrices are embedded with MC and M_6C carbides, as verified by the EDS data

EDS Composition Analysis

- Carbide-forming elements diffuse into carbides as they grow during heat treatment. As diffusion occurs in the heat treatment process, carbide formers migrate into carbides to form the MC, M_2C, and M_6C carbides observed in XRD
- Cr, Mn, Mo, W, V are carbide-formers
- Large, high-strength carbides have higher moduli and can cause weaker matrix to fail at stresses lower than yield stress

Volume Fraction of Carbides and Hardness



- As the hardness increases, the volume fraction decreases which can be seen in the trendline above
  - As carbon is leaving carbides (lower carbide fraction) the carbon is going back into the matrix
- Higher volume fraction of carbides in the annealed conditions for all four alloys tested
- M50 experienced the largest increase of volume fraction percentage from baseline to annealed state (278%)

Hardness Testing

- The baseline heat treated samples showed a higher average hardness than the annealed samples
- Through hardness testing and SEM analysis of the M2 samples, the annealed sample was found to be mislabeled baseline sample, which explains why M2 does not follow the same trend as the other samples

X-Ray Diffraction

- Vanadium–rich MC carbides, molybdenum-chromium M_2C carbides, and tungsten-molybdenum rich M_6C carbides are found
- More carbide peaks are found in annealed samples for M50 and M2, carbides provide wear resistance and strengthening of tool steels. SEM supports these findings too, where carbides are seen to be more evenly distributed

Conclusion

- Annealing decreases hardness and increases volume percentage of carbides.
- A2 carbides show the unique behavior of growing along the grain boundaries during heat treatment, while the other alloys maintain a more uniform distribution.
- Heat treatment changes carbide morphology more than composition.
- MC carbides, M_2C carbides and M_6C carbides are found in the alloy steels.

References

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